VOL. XXXVIII 1920

# TRANSACTIONS

OF THE

# AMERICAN ELECTROCHEMICAL SOCIETY.

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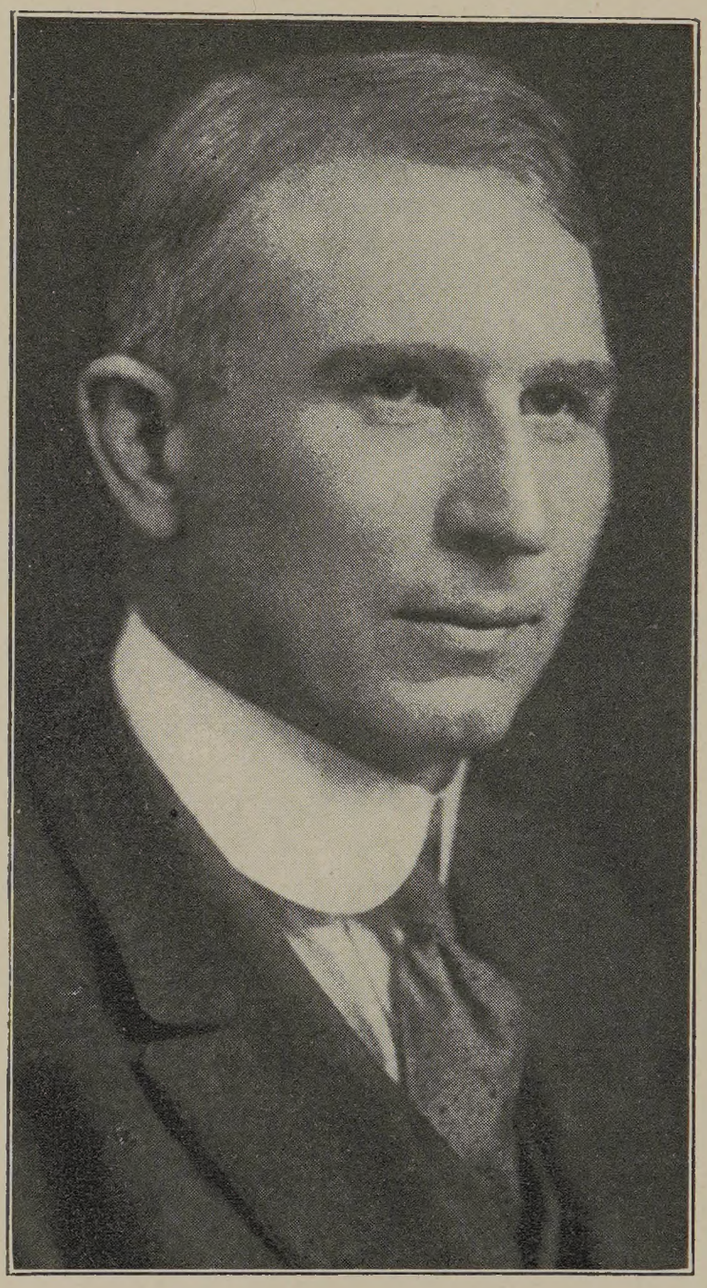

W. S. Landis.

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# TRANSACTIONS

OF THE

# AMERICAN ELECTROCHEMICAL SOCIETY

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VOLUME XXXVIII

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157574

THIRTY-EIGHTH GENERAL MEETING

CLEVELAND, OHIO

SEPTEMBER 30, OCTOBER 1, 2, 1920

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THE AMERICAN ELECTROCHEMICAL SOCIETY

AT THE OFFICE OF THE SECRETARY

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VOLUME XXXVIII 1920

# TRANSACTIONS

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# AMERICAN ELECTROCHEMICAL SOCIETY

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## PROCEEDINGS

CONDENSED MINUTES AND RECORD OF THE THIRTY-EIGHTH GENERAL MEETING OF THE SOCIETY, HELD AT THE HOTEL STATLER, CLEVELAND, OHIO, SEPTEMBER 30, OCTOBER 1 AND 2, 1920.

The attendance at this meeting was 105 members and 80 guests, or a total of 185.

### PROCEEDINGS OF THURSDAY, SEPTEMBER 30, 1920

The President of the Society opened the meeting at 9.30 A. M., and after a few introductory remarks proceeded to the presentation of papers. Papers by the following were considered and discussed: M. R. Wolfe and V. de Wysocki, A. M. Kuhlman and A. D. Spillman, M. L. Hartmann, A. P. Sullivan and D. E. Allen, F. A. J. FitzGerald and Grant C. Moyer, J. W. Richards, E. S. Bardwell, J. Kelleher. These papers are printed in full in these Transactions.

The afternoon was devoted to visiting the following plants: The United States Copper Products Co., the Parish-Pool Co., the Malleable Castings Co., the U. S. Electro-Lead Plating Co. and the Hazelett Storage Battery Co.

At 6.00 P. M. the directors of the Society met in the rooms of the Cleveland Engineering Society, top floor of the Hotel Statler, to hold the regular Board meeting.

At 8.00 P. M. a complimentary smoker, tendered by the National Carbon Co., was held in the ball room of the Hotel Statler. High

grade entertainment features were offered which made the evening very enjoyable.

## PROCEEDINGS OF FRIDAY, OCTOBER 1

A business session preceded the session of reading papers in the Physics Lecture Room, Case School of Applied Science. It was announced that a referendum of the Society as to making the metric system the exclusive legal system of weights and measures of the United States showed 483 in favor to 107 against. A majority of the members present did not favor increasing the annual dues by $1.50 in order that the Society might join the Federated Engineering Societies. The discussion on this item of business follows herewith:

W. S. Landis: The next matter of importance which the Board desires to present to the membership, not for action at this time, but merely to get some further ideas on formulating its own policy in respect to the matter, is the question of the American Electrochemical Society joining the Federation of American Engineering Societies. I do not know how familiar most of you are with this federation, but Mr. Parmelee has been quite active in the matter, and I am going to ask him to tell you what this federation is and how it is made up. Then we will give you some further information from the Society's standpoint of our joining it, and finally we shall open the matter to general discussion. There will be no action taken at this meeting on this matter.

H. C. Parmelee: For a long time men who have been interested in the affairs of technical societies have been concerned as to just what the activities of these various societies should be. Do they fulfill their functions sufficiently when they hold meetings for the discussion of technical papers and perhaps publish them and put a volume in the hands of the members, or is there not something further that a technical engineering society should do? Has it any public relations? Is it advisable to bring engineering opinion to bear on public affairs? Is there any value in having a medium whereby the combined force of engineers and technical men in this country can be expressed when something comes up in which they would have a vital interest? This question was agitated in the large engineering societies first; that is, the American Insti-

tutes and Societies of Civil, Electrical, Mining and Mechanical Engineers, and it resulted in the appointment of a committee of those organizations to consider the aims and purposes and development of those societies; in other words, to find out where they were going and where they ought to go. Those development committees finally recommended that all the engineering societies should get together down in Washington and try and organize some body which would enable engineers to express their opinion on public affairs and participate in public matters and give the country the benefit of engineering and technical advice whenever it was in need of it, the idea being, as I said, that there should be some medium whereby the thought of engineers on public affairs should be articulate. Business men are organized, lawyers are organized, the medical profession is organized; is there any reason for having the engineering and large technical profession disorganized? Consequently, in Washington last June there was organized the Federated American Engineering Societies. The general scheme of it is a society of societies, not of individuals; in other words, this Society, the American Electrochemical Society, can join as an individual; the Mining Engineers, the Electricals, the Mechanicals and the Dallas Technical Society and I think the Cleveland Engineering Society and some other small societies around the country have already joined the Federated American Engineering Societies. The question is whether the American Electrochemical Society wants to join or not. If we do, we will have a delegate or delegates to a governing body known as the American Engineering Council. The American Engineering Council, in turn, would have an Executive Committee which would be the functioning body. Now, if I had known that there was going to be any attempt to make an expression of the purposes of this Society, I could have brought along considerable literature to show what it can do, but it is not the purpose to make any exposition of it, either favorable or unfavorable, but simply to tell you what the scheme of organization is and, in general, its purpose. The purpose I will just read briefly: "The objects of this organization shall be to further the public welfare wherever technical knowledge and engineering experience are involved, and to consider and act upon matters of common concern to the engineering and allied technical professions." Primarily, it is to bring engineering and technical advice

and opinion to bear on public problems. Now, as I say, it is a society made up of other societies. The support of the Federated Engineering Societies comes through dues of $1.50 per member of each member society, and that money will be spent for the objects as I have just read them. Is that sufficient explanation, Mr. President?

W. S. Landis: I think so. A vital thing with respect to our own Society is the question of finances, although there are also questions of policy involved. I am going to ask our Secretary to give us some idea of the financial burden it will place upon us and our ability to meet such burdens.

J. W. Richards: The dues of $1.50 per member, which are put on the national societies which join the Federation, would amount, in our case, to about $3,600 per year. Now we have been keeping our dues to the very minimum; they are only $5.00 per year, that would not enable us to pay this $1.50 per member and come out even at the end of the year. Our margin during the last two or three years has been between $1,500 and $2,000 a year excess of income over expenditures, but the rapidly rising cost of printing has nearly cancelled that this year, so that this year we will about break even; therefore, $1.50 per member could only be raised by adding it to the membership dues. It might be done perhaps voluntarily by asking people to subscribe and having the Society make up the difference for those who did not subscribe, but if it were added to the dues, it would have to be done by amendment to the Constitution. That is the situation. I tried hard to have that amount placed at $1.00 when the committee was considering the dues to be expected from societies, claiming that there would be more societies able to afford $1.00 than $1.50 per member, and that the total received by the Federated Societies would probably be the same, with more people represented; but the large engineering societies decided that $1.50 per member would be the amount. I should say that almost everyone in the Society would say that we should be members of this Federation, but it would mean an addition to the dues of $1.50 per member. I do not see any other way of raising it, and I think it is important that we find out today whether it is the sense of the membership

that we should take a referendum of our whole membership and ask how many would be favorably inclined towards subscribing $1.50 per year extra towards the expenses of this Federation.

W. S. Landis: The Society was invited to appoint a committee to attend this joint organizing conference. That committee was made up of Past-President Bancroft and Mr. Coggeshall. The committee was sent down to the conference merely to sit in as listeners and without power to commit the Society in any way. They reported a majority and minority report, if you can consider such a thing possible in a committee of two, Dr. Bancroft being opposed to our entering the society, while Mr. Coggeshall was at least lukewarmly in favor of joining, possibly a little better than that. I believe Dr. Whitney, himself one of our Past-Presidents, has also written to us stating that he is not in favor of the Society joining the Federated Engineering Societies. Now undoubtedly some of you here have some ideas on this subject, and the Board is looking for help and assistance in the matter, so we will open the meeting to a brief general discussion.

Carl Hering: All I would have said has been said already by Dr. Richards. It simply means that we must then raise our dues $1.50 a year, otherwise we cannot possibly join. I think also that the electrochemists are less interested in the main purposes of this society than engineers, like the electrical, mechanical and civil engineers; the latter are more directly interested in such engineering projects as concern the public welfare as, for instance, the public utilities. We could not take any action until next spring, so we would have plenty of time to consider it. It seems to me that the dues asked are very high and that this Federation is going to have a large amount of money; we would naturally like to know how they are going to spend it. If they are going to spend it on high salaries to a few people, I do not think we ought to contribute so liberally. I have heard some things about their proposed quarters in Washington, and it seems to me they are going to spend money rather lavishly.

W. S. Landis: One of the arguments which struck me as rather interesting was in reference to the fact that the real interest that this Society would have in the Federation would probably lie

largely along the line of advancing waterpower legislation. That is where we would function most intimately with the Government. The answer was made that, acting through our Public Relations Committee, we would probably be quite as effective in Washington, if not more so, than through this Federation. Of course, the finality to this is that if each of us individually went to our nearest Representative we would do a lot more than any federated association of engineering societies along the line of advancing legislation or assisting in Governmental work. I wish the author of those arguments were here so that he could give them to us first hand.

---

After the business session Prof. Dayton C. Miller, Professor of Physics at Case School of Applied Science, gave an illustrated lecture on Sound Phenomena. Professor Miller is probably the most prominent sound physicist in the country, and has done an enormous amount of valuable work in the use of sound phenomena for the location of hidden batteries, as well as in the development of our must refined phonographic apparatus.

Papers by E. A. Keeler, C. J. Rodman and Thos. Spooner, and by J. W. Richards were presented and discussed. They are printed in full in these Transactions.

At 11.30 A. M. a complimentary lunch was served in the gymnasium of Western Reserve University.

The afternoon was devoted to an inspection trip to the Nela Park Laboratories of the General Electric Co. This visit was well planned and proved highly interesting to the participants.

In the evening at 7 o'clock an informal subscription dinner was held in the Ball Room of the Hotel Statler. Dr. A. W. Smith acted as toastmaster and called upon the following for speeches: Dr. Chas. S. Howe, Mr. Walter S. Landis, Mr. Charles F. Brush and Dr. E. P. Hyde.

## PROCEEDINGS OF SATURDAY, OCTOBER 2

Starting at 9.30 A. M., the entire forenoon was taken up with the reading and discussing of papers by the following: A. L.

Ferguson and E. G. Sturdevant, F. C. Mathers and W. H. Bell, Edward F. Kern, F. C. Mathers, F. W. Skirrow and E. R. Stein, E. A. and L. T. Richardson, Wm. D. Richardson. These papers, together with discussions, are printed in full in these Transactions.

After the reading of papers it was announced that in the afternoon visits would be paid to the following plants: Western Reserve Chemical Co., the Ohio Chemical Co. and the Guide Motor Lamp Co. Mr. Burwell, of the Western Reserve Chemical Co., preparatory to visiting his plant, made the following remarks:

Mr. Burwell: It has been announced that the company with which I am connected is making organic chemicals electrolytically. That is not quite true. We restore our solutions by means of electrolytic processes, and then use those solutions for the oxidation of organic bodies to whatever the result may be, such as benzoic acid or anything of that nature. This study was begun about three years ago, as certain of us recognized that it would be necessary, in order to compete with the Germans, eventually, to have at least all the advantages they then had, and among those things that we had no knowledge of in this country, was the reoxidation or the conversion of chromium sulphate to chromic acid. That has been stated in text-books to be a very simple thing. Possibly it is, but we did not find it so. I think Perkins states that the chromium sulphate solutions are flowed through long troughs, and when sufficiently oxidized, are used over again. We have never been able to sufficiently oxidize them without the use of a diaphragm, but we now have a cell that works well at about four volts and has considerable capacity. Also the finding of an anode material that would not fall down was another thing we have done. We are able to reconstruct cells and reconvert chromic sulphate solutions to chromic acid on a commercial scale. We are thus able to convert napthalene into phthalic acid at a cost of less than 14 cents per pound for the product. We have reconverted solutions and used them over 400 times and found no deterioration or ill effect due to the absorption of impurities of any kind, but we have found some rather curious things. I will mention one, and will say beforehand that I do not know why it occurs. In the oxidation of certain organic bodies, such as toluol, to benzoic acid, the reaction becomes so slow after a certain lapse of time,

at the temperature it is commercially possible to use, that you will have left a considerable amount of chromic acid still not reduced. We find that after one restoration (as we call it) and one reoxidation of chromic solution, we can easily send those solutions to "no available oxygen." That is true with all the organic bodies we have used, and that is rather important as, to keep your sulphuric balance in the solution, you must pass the solution first through the cathode and then through the anode compartment, and if you had to reduce and reoxidize, you would lose a large part of your energy efficiency. The selection of the concentration of the solution is an important item. Also we have found that all the products which we make by this means are remarkably pure, and I think that is rather inherent in this form of work. The older forms of oxidation of napthalene do not produce the same purity of product. We had considerable trouble in the sale of our product at first on account of its extreme purity; people refused to use it because they said it must contain a large amount of impurities on account of its high melting point. Eventually we were able to persuade customers that we had the pure product, and today we sell it at 50 percent to 60 percent higher than the product of any other people, and it is in demand all the time; we have never been able to produce enough to fill our orders.

Concluding the session, Dr. Carl Hering presented the following Resolution, which was unanimously adopted:

## RESOLUTION OF THANKS

The American Electrochemical Society, closing its Thirty-eighth Meeting, in Cleveland, wishes to place on record its high appreciation of the unselfish activities and spontaneous hospitality of its members and friends in Cleveland, which have resulted in one of the most enjoyable and profitable meetings held by the Society.

The hearty thanks of the Society are hereby tendered to:

The Directors of the Nela Park Research Laboratories, the National Malleable Castings Company, the United States Copper Products Company, the United States Electro-Lead Plating Company, the Guide Motor Lamp Company, the Electric Steel and Forge Company, the Western Reserve Chemical Company, the

Ohio Chemical Company and the Hazelett Storage Battery Company for opening their plants and laboratories to inspection by our members.

The Authorities of Case School of Applied Science, to Prof. A. W. Smith, and particularly Prof. D. C. Miller, for the use of their building and lecture room for holding a meeting; the Authorities of Western Reserve University for facilities for a luncheon in their Gymnasium.

The National Carbon Company for the very much enjoyed Smoker and Entertainment.

The Cleveland Engineering Society for the hospitable welcome to the use of its rooms, and facilities for lunching.

The Hotel Statler for its accommodation for registration and holding of our scientific sessions, and efficient service in connection therewith.

The Ladies' Committee for their kindly and friendly reception and entertainment of visiting ladies.

And last but not least, the Local Committee and their many co-operating friends for unstinted devotion of time and effort to our comfort and the success of the meeting.

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**January 31, 1921**

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Kohler, H. L.
Nulsen, J. K.
Queeny, J. F.
Teeters, W. R.

**Webb City—**
Wallower, F. C.

### MONTANA

**Anaconda—**
Frick, F. F.
Goodrich, R. R.
O'Brien, A. L.

**Billings—**
Heath, R. F.

**Butte—**
Deshler, G. O.
Naughton, James
Tower, R. E.

**Columbia—**
Mize, M. D.

**Great Falls—**
Bardwell, E. S.
Burns, W. T.
Dow, J. C.
Elton, J. O.
Maclay, E. G.

**Lewiston—**
Hamilton, T. M.

### NEBRASKA

**Lincoln—**
Frankforter, C. J.

**Merriman—**
Kreiss, A. L.

**Omaha—**
Hall, A. E.
O'Hara, B. M.
Randall, A. G.
Reinbold, Herman
Reinbold, Hugo

**Valley—**
Whitmore, J. D.

### NEVADA

**Reno—**
Lind, S. C.

**Searchlight—**
Newport, F. T.

### NEW HAMPSHIRE

**Berlin—**
Barton, C. B.
Comstock, R. L.
Fogarty, J. A.
McMurtrie, D. H.
Moore, H. K
Richter, G. A.

**Durham—**
Perley, G. A.

**Wilton—**
Abbott, Wm. G., Jr.

### NEW JERSEY

**Arlington—**
Pickering, O. W.
Proctor, C. H.

**Asbury Park—**
Fernberger, H. M.
Wooley, W. R.

**Atlantic City—**
Rhodin, B. E. F.

**Bayonne—**
Bjorkstedt, Wm.
Corse, W. M.
Crawford, C. A.
Kendrick, T. E.
Mahlman, O. L.
Merica, P. D.

**Bayway—**
Burgess, Louis

**Bloomfield—**
Amer, H. S.
Braley, H. D.
Hageman, A. M.
Kleinfeldt, H. F.
McAllister, Paul
Major, Ralph M.
Romanelli, E.
Vassar, H. S.

**Boonton—**
Seabury, R. W.

**Bordentown—**
Mulligan, J. J.

**Bound Brook—**
Dailey, J. G.
Saunders, H. F.

**Butler—**
Buttfield, A. C.

NEW JERSEY—Continued

**Carneys Point—**
Lee, I. E.

**Chrome—**
Clark, J. C.
Deacon, R. W.
Green, H. M.
Greenwood, H. D.
Haeseler, Paul C.
Hood, B. B.
Merrill, M. W.

**Cranford—**
Lake, D. B.
Taber, H. P.
White, J. H.

**Dover—**
Pickett, O. A.
Struthers, J. A.

**Eaglewood—**
Fensterer, W. J.

**East Orange—**
Cowles, Harry D.
Driver, W. B.
Honan, E. M.
Mommo, E. J.
Prindle, E. J.

**Edgewater—**
Grille, G. A., Jr.

**Elizabeth—**
Dimock, W. P.
Little, W. T.
Martens, Paul
Miller, A. B.
Pyne, F. R.
Schonberg, J. R.
Treacy, C. S.

**Englewood—**
Edwards, W. F.

**Essex Falls—**
Liebschutz, Morton
Robb, Chas. D.

**Glen Ridge—**
Cox, H. N.

**Gloucester City—**
Knoedler, E. L.
Miner, H. S.

**Haddon Heights—**
Meyer, John

**Harrison—**
Needham, H. H.

**Haskell—**
Cummings, Wm. J.

**High Bridge—**
Berlin, W. G.
Hall, John H.
Le Boutillier, Clement

**Hoboken—**
Farnsworth, Frank
Hazeltine, L. A.
Winninghoff, W. J.

**Irvington—**
Bachofner, D. K.

**Jersey City—**
Baldwin, A. T.
Logan, M. H.
Lubowsky, Simon J.
Porth, H. W.
Swartley, H. R., Jr.
Wilson, W. K.
Zeller, Richard

NEW JERSEY—Continued

**Kenvil—**
Schantz, J. M.

**Keyport—**
Schwarz, Edw.

**Lawrenceville—**
Willcox, Dudley

**Leonia—**
Folsom, R. A.
Holler, H. D.

**Maplewood—**
Cunningham, F. W.
Wright, J. C.

**Maywood—**
Manwaring, F. G.

**Merchantville—**
Wickes, C. S.

**Millstone—**
Ruh, C. H.

**Montclair—**
Bradley, Linn
Crane, F. D.
Ellis, Carleton
Eurich, E. F.
Gesell, W. H.

**Morristown—**
Anger, E. M.
Mandell, A. J.

**Newark—**
Barker, P.
Benjamin, E. O.
Boice, E. N.
Carter, F. E.
Ceader, Jos.
Colby, E. A.
Gifford, W. E.
Hart, L. O.
Holding, H. H.
Hubley, W. F.
Keen, Wm. H.
Marlatt, C. D.
Maywald, F. J.
McNeill, Ralph
Prochazka, J. A.
Robinson, F. W.
Sievering, Philip
Vorce, L. D.
Walsh, P. C., Jr.
Weber, M. G.
Weston, E.
Youry, W. H. S.

**New Brunswick—**
Clark, C. B.
Keller, Oran
Richardson, L. T.
Voorhees, L. A.

**Newfoundland—**
Frederick, Geo. E., Jr.

**Nutley—**
Richey, Wm. A.

**Ocean Grove—**
Bergen, C. R.

**Orange—**
Edison, T. A.
Kammerhoff, H. H.
Sholes, C. E.

**Parlin—**
Buchanan, A. E., Jr.
Pitman, E. C.
Small, J. O.

**Paterson—**
Jarvis, E. G.

**NEW JERSEY—Continued**

**Perth Amboy—**
Antisell, F. L.
Fisher, H. W.
Hume, Neil
McNitt, R. J.
Peakes, G. L.
Peck, E. B.
Rasmussen, F. J.
Roessler, F.
Rossi, Louis M.
Skowronski, S.
Stewart, M. E.
Waring, T. D.

**Phillipsburg—**
Baker, John T.

**Plainfield—**
Hibbard, H. D.
Manahan, Paul R.
Sheffield, W. T.
Spicer, C. W.

**Princeton—**
Furman, N. H.
Heisig, G. B.
Hulett, G. A.
Northrup, E. F.
Smith, D. P.
Taylor, H. S.

**Rahway—**
Maeulen, Frederick
Murray, B. L.

**Red Bank—**
Moore, C. W.

**Ridgewood—**
Czarnecki, F. C.

**Roselle—**
Grymes, E. S.

**Rutherford—**
Schroeder, C. M. E.

**Sewaren—**
Buttfield, W. J.
Cowles, A. H.

**Short Hills—**
Hough, Arthur

**South Orange—**
Bajda, Jas. J.
Yunck, Carl I.
Yunck, J. A.

**Summit—**
Hornsey, J. W.

**Trenton—**
Harter, Wickham
Pardoe, W. D.
Porter, H. F.
Saums, H. L.

**Upper Montclair—**
Buzby, A. D.

**Watchung—**
Moldenke, Richard

**Weehawken—**
Kraus, Ernest
Laise, C. A.
Schubert, B. H.

**Westfield—**
Alexander, H. H.

**West Orange—**
Pedersen, A. Z.
Worth, B. G., Jr.

**Wood Ridge—**
Carrier, S. C.

**NEW MEXICO**

**Carrizozo—**
Wright, J. E.

**Santa Fe—**
Grubnau, G. M.

**Tyrone—**
Lyons, C. F.

**NEW YORK**

**Albany—**
Adams, J. F.
Daft, Leo
Paulsson, Axel
Pulman, O. S.

**Amagansett—**
Leak, N. L.

**Astoria—**
Bernabo, Louis
Lewis, C. A.

**Auburn—**
Case, T. W.

**Barnard—**
Schneider, Louis

**Batavia—**
Walker, E. C., 3d

**Bedford Hills—**
Howe, H. M.

**Beechurst (L. I.)—**
Landis, W. S.

**Blasdell—**
Harty, W. A.

**Brooklyn—**
Altenberg, Leo
Behnken, H. E.
Burger, Alfred
Cecere, T. J.
Cowan, W. A.
Dorsam, W. C.
Edwards, I. W.
Erhart, W. H.
Foster, O. R.
Gumaer, P. W.
Harkins, W. D.
Harkness, W. E.
Honeyman, R. B., Jr.
Hooker, S. C.
Keenan, T. J.
Landolt, P. E.
Lang, Franklin
Lodyguine, A.
Mahoney, J. N.
Pack, Chas.
Schultz, F. H.
Scott, L. C.
Sperry, E. A.
Taylor, F. D.

**Buffalo—**
Albright, L.
Bierbaum, C. H.
Carrier, W. H.
Cushing, H. M.
Doty, E. L.
Grotzinger, John
Harrington, R. M.
Jones, W. B.
Kempke, C. W.
Knox, L. B.
Loncteaux, L. W.
McBerty, Ford H.
Neal, J. R. H.
Patch, N. K. B.
Rahill, E. J.

NEW YORK—Continued

**Buffalo—Continued**
Read, Wm. C.
Redfield, C. A.
Rippel, E. G.
Ruthenburg, Marcus
Sicard, H. C.
Stephenson, H. L.
Underwood, C. W.
Van Marle, D. J.
Ward, H. Lee
Wilke, Wm.
Wood, C. H.
Yager, J. J.
Zaremba, Edward

**Canandaigua—**
Phillips, Ross
Weeks, F. D.

**Clinton—**
Saunders, A. P.

**Corning—**
Hood, H. P.
Kelsey, V. V.
Sullivan, E. C.

**Croghan—**
Sisson, G. H.

**Douglaston (L. I.)—**
Vom Baur, C. H.

**Dunkirk—**
Lounsberry, F. B.

**Elmira—**
Carrier, C. F., Jr.

**Flushing (L. I.)—**
Kemper, D. A.
Watson, P. B.

**Fort Plain—**
Fox, W. J.

**Herkimer—**
Patterson, C. T.

**Ithaca—**
Bancroft, Wilder D.
Barrett, W. R.
Gillett, H. W.
Milligan, L. H.

**Jamaica—**
Brons, H. F.
De Fries, H. A.

**Kenwood—**
Reeve, A. G.

**LaSalle—**
Allen, D. E.
Gilbert, H. N.
Wilson, N. A.

**Lawrence (L. I.)—**
Guiterman, K. S.

**Little Falls—**
Tinkler, L. G.

**Lockport—**
Howard, L. E.
Kenan, W. R., Jr.
Neal, J. B.

**Long Island City—**
Birdsall, C. G.
Reed, D. C.
Rees, R. R.

**Lyon Mountain—**
Brakes, James

**Massena—**
Doerschuk, V. C.
Mantel, Chas.
Whitaker, A. W., Jr.
Wilmore, C. B.

NEW YORK—Continued

**Monticello—**
von Isakovics, Mary U.

**Mt. Vernon—**
Dennis, R. E.
Wilson, C. H.

**New Brighton, S. I.—**
Walter, H. W.

**New York City—**
Abbé, P. O.
Acheson, E. G.
Adamson, G. P.
Addicks, L.
Aldridge, W. H.
Alley, J. C.
Alley, W. R.
Allyn, R. S.
Atwater, C. G.
Barnes, H. H., Jr.
Barrows, F. E.
Barstow, W. S.
Bateson, C. E. W.
Bauer, A. D.
Beck, E. A.
Bedell, E. H.
Blossom, E. L.
Bogue, C. J.
Bowman, W.
Brindley, Geo. F.
Brown, H. P.
Browne, deC. B.
Brumberg, Geo.
Buch, N. W.
Buck, H. W.
Buck, L. J.
Buckie, R. H.
Bull, I. C.
Bump, M. R.
Cameron, W. S.
Cardoza, J. B. N.
Carse, D. B.
Chandler, C. F.
Chappell, H. F.
Clark, W. G.
Clark, W. J.
Coho, H. B.
Cohoe, W. P.
Colcord, F. F.
Cone, E. F.
Cooper, K. F.
Corning, C. R.
Cunniff, John
De Geer, Gerard
Deppe, W. P.
Devorken, Harry
Doerfinger, W. F.
Doremus, C. A.
Dorr, J. V. N.
Dreyfus, W.
Duce, J. F.
Dutton, W. C.
Dyrssen, Waldemar
Eagle, H. Y.
Eckelmann, L. E.
Eimer, A.
Englehard, C.
Featherstone, W. B.
Field, Crosby
Fink, Colin G.
Fish, Job, Jr.
Fitting, R. U.
Fitzgerald, W. V.
FitzGibbon, R.
Fleming, R.
Ford, F. E.

**NEW YORK—Continued**

**New York City—Continued**

Frank, K. G.
Fries, H. H.
Fukuzawa, T.
Gaines, R. H.
Glenn, E. R.
Goepel, C. P.
Gray, J. H.
Green, R. deC.
Grosvenor, W. M.
Halcomb, C. H.
Hale, E. W.
Hambuechen, Carl
Hampton, W. H.
Hansell, N. V.
Harris, J. W.
Hatzel, J. C.
Havill, O. A.
Hendrie, G. A.
Hendry, W. F.
Herty, C. H.
Hill, H. P.
Hill, N. S., Jr.
Hirsch, Alcan
Hirschland, F. H.
Hoge, J. F. D.
Hotchkiss, F. H.
Hunt, A. M.
Huntoon, L. D.
Hurum, F.
Iliff, J. W.
Ingalls, W. R.
Iwata, Hiroshi
Jacobson, B. H.
Jacoby, H. E.
Jicha, John
Jordon, W. L.
Jorgensen, E. L.
Junqueira, S. M.
Kahn, Julius
Keller, Edw.
Kennedy, J. J.
Kern, E. F.
Kingsley, E. D.
Klipstein, E. C.
Knapp, Geo. O.
Kohn, M. M.
Kunz, G. F.
Langmuir, A. C.
Leavitt, Wm. F. B.
Ledoux, A. R.
Lepsöe, Robert
Liebmann, A. J.
Lloyd, R. McA.
Loebell, H. O.
Lovejoy, D. R.
MacDonald, J. A.
Madsen, C. P.
Magnus, Benjamin
Maier, C. G.
Mailloux, C. O.
Manning, P. D.
Mantius, Otto
Marsh, C. W.
Martin, T. C.
Marvin, A. B.
Mason, F. S.
Mastick, S. C.
Mathewson, E. P.
Mays, S. W.
McDaniel, A. S.
Mershon, R. D.
Metz, G. P.
Metz, H. A.
Metzger, F. J.

**NEW YORK—Continued**

**New York City—Continued**

Miller, B. H.
Miller, D. D.
Moody, H. R.
Morehead, J. M.
Morgan, J. L. R.
Morrison, A. C.
Mortimer, J. D.
Mueser, E. E.
Muir, J. M.
Muschenheim, F. A.
Myers, W. S.
Nakano, Y.
Nichols, W. H.
Nichols, W. S.
Norton, W. S.
O'Neill, W. J.
Onoda, N. P.
Parmelee, H. C.
Pennie, J. C.
Petinot, N.
Pranke, Edw. J.
Price, E. F.
Prosser, H. A.
Raibourn, P. A.
Raimondo, Sebastiano
Randall, J. W. H.
Reeds, S. A.
Reed, S. A.
Reid, G. H.
Reno, J. W.
Ricketts, L. D.
Riglander, M. M.
Riker, J. J.
Riley, L. A., 2d
Roller, F. W.
Ross, Elbert
Roth, C. F.
Ruffner, C. S.
Ruhl, L.
Ruhm, H. D.
Sachs, A. P.
Sargent, G. J.
Schildt, H. R.
Schloss, J. A.
Schuetz, F. F.
Schuyler, A. H.
Scott, E. K.
Seward, G. O.
Sharp, C. H.
Shimada, M.
Simon, C. K.
Singer, C. E.
Smith, Dyer
Smith, E. A. Cappelen
Snook, H. C.
Southgate, G. T.
Sowers, Ossa
Spandow, W. E.
Speed, Buckner
Speiden, C. C.
Speirs, Chas. E.
Spicer, H. N.
Starr, H. W.
Statham, Noel
Stevenson, Reston
Stockelbach, F. E.
Stoughton, Bradley
Summers, L. L.
Swenarton, W. H.
Takamine, J.
Takamine, J., Jr.
Tate, A. O.
Teeple, J. E.
Thatcher, C. J.

NEW YORK—Continued

**New York City—Continued**

Toch, M.
Tucker, S. A.
Waddell, M.
Wadleigh, F. R.
Walker, A. L.
Walker, G. E.
Walker, R. G.
Ward, A. T.
Warner, F. H.
Washburn, F. S.
Waterman, F. N.
Weber, J. E.
Weintraub, Ezechiel
Westervelt, W. Y.
Wheeler, A. E.
Whitaker, M. C.
White, J. G.
Wigglesworth, H.
Williams, A.
Williams, L. W.
Winship, W. W.
Witherell, C. S.
Wolf, H. J.
Wright, Arthur
Zons, F. W.

**Niagara Falls—**

Acheson, E. G., Jr.
Avery, J. M.
Bakken, H. E.
Baldwin, R. L.
Barton, P. P.
Becket, F. M.
Bishop, Robert
Bliss, Wm. Lord
Brallier, P. S.
Brown, R. G.
Campbell, Geo.
Carveth, H. R.
Case, F. P.
Chace, R. T.
Cole, E. R.
Converse, V. G.
Cox, G. E.
Critchett, J. H.
Downs, J. C.
Dunlap, O. E.
Edmands, I. R.
Field, A. L.
Field, B. E.
FitzGerald, F. A. J.
Fowler, R. E.
Fuller, G. P.
Gailey, A. J.
Gegenheimer, R. E.
Glaze, John B.
Griffith, J. R.
Guinther, John
Hamann, A. M.
Hardie, C. G.
Harper, J. L.
Hartmann, M. L.
Harvey, W. G.
Hauman, E. L.
Herzog, G. K.
Hills, L. H.
Hinckley, A. T.
Hinman, R. G.
Hix, Edward L.
Hooker, A. H.
Houser, C. S.
Hutchins, Otis
Imlay, L. E.
Johnson, J. A.
Judson, L. C.

NEW YORK—Continued

**Niagara Falls—Continued**

Kellogg, H. W.
Kemmer, F. R.
Koethen, F. L.
Kokatnur, V. R.
Lansing, C. N.
Lavene, H. A.
Lee, H. R.
Lidbury, F. A.
Low, F. S.
Lyster, T. L. B.
MacMahon, J. D.
MacMillan, J. R.
Marshall, J. G.
Mauran, M.
McKnight, W. A.
McMillen, Herbert
Meredith, W. F.
Miller, D. R.
Moritz, C. H.
Morley, M. H.
Moyer, G. C.
Muggleton, G. D.
Noyes, H. L.
Nutting, E. G.
Oatman, A. B.
Ohliger, L. P.
Osborne, S. E.
Patterson, L. G.
Patterson, T. A.
Plock, A. F.
Ross, Wm.
Rossi, A. J.
Rowland, J. M.
Rykenboer, E. A.
Savage, P. G.
Schoellkopf, P. A.
Sergeant, E. M.
Sims, C. E.
Simmers, A. L.
Smith, Acheson
Smith, E. S.
Snowdon, R. C.
Speiden, E. C.
Stamps, F. A.
Stillesen, J. M. A.
Stone, G. W.
Tone, F. J.
Turnbull, Robert
Udy, M. J.
Uptegraff, T. M.
Vaughn, C. F.
Wallace, W.
Walworth, S. L.
Wells, H. P.
Wescott, E. W.
White, H. E.
White, R. H.
Williamson, A. M.
Yngve, Victor

**Nyack—**

Betts, A. G.

**Oneida—**

Bailey, R. O.
Shartzer, E. S.

**Pelham—**

Brown, C. J.

**Penn Yan—**

Taylor, C. E.

**Port Richmond (S. I.)—**

Strauss, F. A.

**Potsdam—**

Roess, Harold
Russell, L. K.

NEW YORK—Continued

**Prince Bay (L. I.)—**
Johnston, F. A.

**Richmond Hill (L. I.)—**
Haslwanter, C.
Herreshoff, J. B., Jr.
Koch, G. W.

**Rochester—**
Burch, W. S.
Miller, S. W.
Seger, G. E.
Sheppard, S. E.
Will, R. T.

**Schenectady—**
Allen, O. F.
Andrews, Mary R.
Andrews, W. S.
Arsem, W. C.
Berg, E. J.
Coffin, F. P.
Collins, E. F.
Coolidge, Wm. D.
Creighton, E. E. F.
Dantsizen, Christian
Dushman, Saul
Hansen, C. A.
Hawkins, L. A.
Hilliard, J. D.
Isshiki, T.
Kienle, R. H.
Langmuir, I.
Lof, E. A.
McTerney, P. A.
Murphy, E. J.
Reist, H. G.
Rice, E. W., Jr.
Rushmore, D. B.
Seede, J. A.
Steinmetz, C. P.
Taylor, J. B.
Thurston, L. S.
Van Brunt, C.
Whitney, W. R.
Willey, L. M.
Winne, H. A.
Zeh, G. G.

**Sherrill—**
Thurber, A. E.

**Stony Point—**
Hood, H. P.

**Syracuse—**
Bellinger, H. P.
Berry, G. M.
Brookfield, W. B.
Conklin, E. B.
Handy, B. H.
Houghton, A. C.
Moore, E. T.
Newkirk, E. D.
Pearce, W. C.
Pennock, J. D.

**Tottenville (S. I.)—**
Temple, Sterling
Wagner, C. S.

**Troy—**
Albright, P. S.
Beliaeff, S.
Bryson, T. A.
Hunter, M. A.
Jones, Augustus
Lincoln, A. T.
Matyas, J. R.
Rosenholtz, J. L.
Voorhies, J. A.

NEW YORK—Continued

**Tuckahoe—**
Elbert, J. J.

**Utica—**
Putnam, G. I.
Steele, F. B.

**Watertown—**
Wilder, M. S.

**Watervliet—**
Dudley, Boyd, Jr.

**Yonkers—**
Baekeland, L. H.
Duurloo, F.
Eldridge, C. H.
Morey, S. R.

NORTH CAROLINA

**Badin—**
Parks, R. E.

**Charlotte—**
Gilchrist, P. S.
Lee, W. S.

**Clemmons—**
Pickens, Rufus H.

OHIO

**Akron—**
Knight, M. A.
Shaw, E. C.

**Alliance—**
Baily, T. F.
Cope, F. T.

**Barberton—**
Austin, A. O.
Lyons, H. N.

**Berlin Heights—**
Jenkins, L. S.

**Canton—**
Buckwalter, T. V.
MacGregor, Walter

**Chagrin Falls—**
Burwell, A. W.

**Cincinnati—**
Bray, C. A.
Dittmar, Carl
Ecker, Howard, Jr.
Elliott, Geo. K.
Hughes, J. S.
Pugh, A. H., Jr.

**Cleveland—**
Arnold, E. A.
Barron, A. N.
Blake, H. D.
Blouch, H. H.
Booth, H. S.
Chaffe, J. B., Jr.
Clymer, W. R.
Evans, L. L.
Fahrenwald, F. A.
Fleming, S. H.
Foster, C. L.
Frank, E. C.
Gardner, J. H.
Goss, B. C.
Graves, W. G.
Hamister, V. C. H.
Hasslacher, Jacob
Hemingway, Frank
Herron, J. H.
Howard, Henry
Hyde, E. P.

**OHIO—Continued**

**Cleveland—Continued**

Koehler, W.
Kranz, W. G.
Laib, Walter
Lapidos, R. H.
Lincoln, P. M.
Marshall, G. G.
Maschke, Otto
McIntosh, H. G.
Megroot, J. P.
Merrill, G. S.
Morgan, H. J.
Munn, J. C.
Pratt, E. B.
Reinhardt, W. L.
Richardson, E. A.
Saunders, E. H.
Smith, A. W.
Tower, O. F.
Wallau, H. L.
Weidenthal, H. G.
Whitlock, E. H.
Wilcox, W. G.
Woodward, J. M.

**Columbus—**

Coe, E. R.
Demorest, D. J.
French, E. H.
Lower, J. R.
Moormann, T. A.
Schaaf, Downs
Withrow, J. R.

**Dayton—**

Clements, F. O.
Fox, C. P.
Hummert, R. H.
May, R. B.
Niswonger, E. E.
Williams, H. M.

**East Palestine—**

Owen, F. E.

**Elyria—**

Stark, E. E.

**Fostoria—**

Downes, A. C.

**Fremont—**

Goodwin, J. H.

**Galion—**

McBerty, F. R.

**Granville—**

Freas, F. B.

**Lakewood—**

Bullock, A. R.
Chaney, N. K.
Chase, W. S.
Drefahl, L. C.
Gillingham, C. A.
Hazelett, C. W.
Krejci, M. W.
Kuhn, H. M.
McQuaid, H. S.
Mott, W. R.
Ordway, D. L.
Reid, R. R.
Russell, C. A.
Schmidt, J. H.
Waitt, W. G.

**Mansfield—**

Scott, W. S.
Wolf, F. L.

**OHIO—Continued**

**Middletown—**

Ahlbrandt, G. F.
Aupperle, J. A.
Beck, W. J.
Eldridge, S. E.

**Napoleon—**

Long, G. E.

**Norwood—**

Bell, Wm. H.

**Portsmouth—**

Wooldridge, W. J.

**Ravenna—**

Mann, W. W.

**Salem—**

Davis, D. L.
Eberwein, S. J.

**Springfield—**

Ring, C. W.

**Toledo—**

Ensign, R. W.
Moorhouse, L. B.
Nagel, W. G.
West, P. A.

**Wadsworth—**

Shapiro, H.

**Youngstown—**

McKeown, Wm.
Russell, D. A.

**OKLAHOMA**

**Bartlesville—**

Mize, M. D.

**Muskogee—**

Born, Sidney

**Norman—**

Williams, G. Y.

**Tulsa—**

Cleage, T. D.
Higgins, E. C., Jr.
Kroll, Cornelius
Miller, Walter
Taber, G. H., Jr.

**OREGON**

**Athena—**

Bennett, W. E.

**Corvallis—**

Rowland, F. E.

**Grands Pass—**

Corliss, H. A.

**Parkdale—**

Slaten, F. W.

**Portland—**

Hall, E. L.
Morrison, W. L.
Oldright, G. L.
Strong, R. K.

**PENNSYLVANIA**

**Allentown—**

Snelling, W. O.

**Altoona—**

Casselberry, H.

**Ambridge—**

Meineke, O. H.

**Arnold—**

Forman, L. P.

PENNSYLVANIA—Continued

**Aspinwall—**
Hedden, S. E.
Hessom, B. F., Jr.
McMillen, R. H.
O'Donovan, J. S.

**Beaver—**
Dougherty, J. W.

**Berwyn—**
Scholl, W. D.

**Bethlehem—**
Buck, C. A.
Burgess, C. W.
Butts, Allison
Fehnel, J. Wm.
Hommel, R. P.
Kamura, H.
Kay, M. J.
Lehr, H. D.
Luckenbach, L. J.
MacNutt, Barry
Mumma, P. F.
Pulsifer, H. B.
Richards, J. W.
Roush, G. A.
Salmon, C. P.
Seyfert, S. S.
Smull, J. G.
Spurrier, J. R.
Uihlein, R. A.

**Brackenridge—**
Connell, H. R.
Ober, J. E.

**Braebun—**
Schilling, H. A.

**Bridgeville—**
Anderson, A. N.
Saklatwalla, B. D.

**Canonsburg—**
Nicholson, K. C.
Vogt, L. F.

**Charleroi—**
Eakin, C. T.
Grossman, M. A.

**Chester—**
Comey, A. M.
Krause, W. B.

**Conshohocken—**
Davis, F. W.

**Coraopolis—**
Hensen, Emil
Stafford, S. G.

**Creighton—**
Cox, S. F.
Gelstharp, F.

**Cynwyd—**
Parkhurst, C. W.

**Duquesne—**
Burroughs, D. C.
Cummins, A. C.
Rockoff, A. A.
Stewart, H. M.

**Easton—**
Gordon, C. McC.
Hart, E.
Mack, H. F.
Shimer, E. B.

**Ellwood City—**
Dunn, J. J.
Offutt, J. W.

PENNSYLVANIA—Continued

**Emaus—**
Sefing, F. G.

**Erie—**
Valentine, I. R.

**Gettysburg—**
Parsons, L. A.

**Glen Loch—**
Rittenhouse, E.

**Greensburg—**
Koppitz, C. G.

**Grove City—**
Welker, J. A.

**Harrisburg—**
Kinter, G. R.
Smith, J. H.

**Holtwood—**
Bang, A. F.

**Irvin—**
Clark, K. G.

**Jenkintown—**
Davis, R. W., Jr.

**Lancaster—**
Faber, H. B.

**Lansdowne—**
Kent, S. L.

**Latrobe—**
Garratt, Frank
Gill, J. P.
Sherk, H. C.

**Lebanon—**
Weimer, E. A.

**Mars—**
Adair, W. R.

**McKeesport—**
Goodspeed, G. M.

**Media—**
Brooke, F. W.

**Milton—**
Forrester, W. R.

**Monessen—**
Owens, E. W.

**Mount Airy—**
Hall, C. A.

**Mount Penn—**
Esterly, J. E.
Kramer, L. B.

**Mount Union—**
Harvey, F. A.

**Munhall—**
Hopkins, G. A.

**Natrona—**
Darlington, H. T.
Huff, E. L.

**New Kensington—**
Beatty, H. H.
Bell, A. D.
Blough, Earl
Churchill, H. V.
Hamor, W. A.

**Oakmont—**
Frary, F. C.
Moore, J. R.

**Oil City—**
Bayless, F. B.

**PENNSYLVANIA—Continued**

**Palmerton—**
Breyer, F. G.
Holstein, L. S.
Vanderwaart, P. T.

**Parkesburg—**
Beale, H. A.

**Philadelphia—**
Asef, Waldemar,
Atkinson, Wm. W.
Bash, F. E.
Bassett, H. P.
Baush, J. R.
Benoliel, S. D.
Blasius, C. E.
Bonine, Chas. E.
Brophy, O.
Brown, R. P.
Callen, A. S.
Canfield, J. M., Jr.
Chillas, R. B.
Clamer, G. H.
Cooney, E. R.
Corbin, J. Ross
Dalton, Walter
Devereux, W.
Eglin, W. C. L.
Fletcher, W. E.
Foglesong, J. E.
Fulweiler, W. H.
Furness, R.
Gellert, N. H.
Gersen, F. C.
Gibbs, A. E.
Gould, D. F.
Graham, A. K.
Guest, I. S.
Haig, J. E.
Harrison, W. E.
Hering, Carl
Hess, H.
Hitchcock, F. R. M.
Hodson, Frank
Holland, W. E.
Howard, G. M.
James, W. F.
Keith, N. S.
Koons, M. R.
Kummer, M. T.
Kutz, Milton
Lafore, J. A.
Laird, K. V.
Lavino, E. J.
Lay, J. T.
Levin, I. H.
Lucas, C. C., Jr.
Lukens, H. S.
Mahlman, O. L.
Mainwaring, W. D.
McConnell, J. Y.
Meigs, C. C.
Merzbacher, Aaron
Moerk, F. N.
Mottinger, B. T.
Neville, Neil
Oldach, F. W.
Paul, H. N.
Peirson, C. L.
Reich, J. S.
Rosenfeld, J. R.
Rosengarten, G. D.
Ross, E. S.
Russell, C. J.
Ryan, F. J.
Sadtler, S. P.
Sadtler, S. S.

**PENNSYLVANIA—Continued**

**Philadelphia—Continued**
Salom, P. G.
Schamberg, M.
Scott, W. M.
Short, Frank
Smith, E. F.
Smith, E. W.
Smith, J. B.
Stevens, J. F.
Styri, Haakon
Taggart, W. T.
Taylor, H. N.
Thompson, C. M., Jr.
Thwing, C. B.
Toll, J. D.
Weisenburg, Andrew
Williams, H. J.
Wirt, Chas.

**Pittsburgh—**
Aston, James
Bacon, R. F.
Blakeslee, D. W.
Brown, John T., Jr.
Brownlee, R. H.
Buck, D. M.
Caplan, S. J.
Carpenter, C. H.
Clarke, E. B.
Crabtree, F.
Darrin, Marc
Dunn, H. E.
Edgerton, C. T.
Eschholz, O. H.
Gadsby, Geo. M.
Gibson, C. B
Goodale, S. L.
Graef, W. H.
Graybill, J. H.
Hartley, R. H.
Hecker, Chas.
Higgins, D. F.
Hitchcock, H. K.
Hoover, P. L.
Imhoff, W. G.
Jacoby, H. M.
James, J. H.
Jones, G. W.
Kemery, P.
Kier, S. M.
Laughlin, H. H.
Lindsay, W. H.
Loeffler, Geo. O.
MacRae, Duncan
Mathias, D. L.
McDonald, R. A.
McKinley, Jos.
McNiff, G. P.
Mathews, J. A.
Meyers, H. H.
Moore, R. W. E.
Moore, W. E.
Nairn, J. M.
O'Neil, R. D.
Page, G. S.
Palmer, Chas. S.
Pinkerton, A.
Pope, Chas. E.
Robbins, Chas.
Rodman, C. J.
Schluederberg, C. G.
Schoepf, T. H.
Silverman, A.
Simmons, C. W.
Skinner, C. E.
Slocum, F. L.

**PENNSYLVANIA—Continued**

**Pittsburgh—Continued**
Snyder, C. G.
Speller, F. N.
Staley, F. R.
Stone, E. C.
Tafel, Theodore, Jr.
Taylerson, E. S.
Turnock, E. Hill, Jr.
Turnock, L. C.
Uhlenhaut, F., Jr.
Unger, J. S.
Viol, C. H.
Weidlein, E. R.
Wilcox, E. A.
Wiley, B.
Wilson, I. W.
Yardley, J. L. McK.
Zimmerman, R. E.

**Primos—**
Boericke, G.

**Pulaski—**
McCullough, H. F.

**Reading—**
Miller, Daniel

**Roaring Springs—**
McDonald, F.

**Scranton—**
Winkel, Herbert

**State College—**
Armstrong, H. M.
Chedsey, W. R.
Knight, O. A.
Soley, W. G.

**Steelton—**
Reed, J. C.

**St. Marys—**
Sullivan, A. P.
Wilson, G. C.

**Swarthmore—**
Alleman, G.
Creighton, H. J.
Sackett, B. R.

**Swissvale—**
Grondahl, L. O.
Lewis, J. D.
Marden, J. W.

**Tamaqua—**
Brown, K. R.
Long, R. A.

**Tarentum—**
Parkinson, J. C.

**Titusville—**
Evans, C. T.

**Tyrone—**
Woodbridge, W. B.

**Uniontown—**
Fleming, H. P.

**Verona—**
Rodman, Hugh

**Washington—**
Arnold, H. C.
Duval, A. L.
Sargent, G. W.
Weitzenkorn, J. W.

**Wilkinsburg—**
Hewitt, A. C.
Hineline, H. D.
Johnson, J.
Lewis, J. W.
Ross, E. E.
Wills, W. H., Jr.

**PENNSYLVANIA—Continued**

**Wyomissing—**
Parker, J. H.

**York—**
Bleakley, W. Y.
Fineman, Carl
Smith, Lyon

## RHODE ISLAND

**Newport—**
Eppley, Marion

**Providence—**
Andrews, J. H.
Chambers, R. F.
Nagelvoort, Adriaan
Saunders, W. M.
Thurber, J. H.
Willson, Ellery S.

## SOUTH CAROLINA

**Charleston—**
Hughes, H.

**Columbia—**
Mills, J. E.

**Sumter—**
Van Deventer, H. R.

## TENNESSEE

**Alcoa—**
Glascock, B. L.

**Bristol—**
Hiddleson, W. A.

**Chattanooga—**
Davison, G. L.
Kruesi, P. J.
Waldo, W. G.
Wiggins, C. Q.

**Kingsport—**
White, J. F.
Whitnall, R. T.

**Knoxville—**
Mitman, Wm. T.
Sammons, W. R.

**Maryville—**
Swany, G. R.

**Mascot—**
Ammon, Robert

**New Market—**
Livingston, W. S., Jr.

**Signal Mountain—**
Taylor, W. W.

## TEXAS

**Austin—**
Schoch, E. P.
Walker, T. B.

**El Paso—**
Fraser, Lee
Schimerka, F. S.

**Fort Worth—**
Eastman, H. M.

**Houston—**
Henst, J. Vander
Kroemer, F. W.
Weiser, H. B.

**Palestine—**
Hunt, N. H.

**TEXAS—Continued**

**Port Arthur—**
Roberts, G. I.
Slater, W. A.

**San Antonio—**
Williamson, P. B.

**Waxahachie—**
Alexander, C. M.

**Wichita Falls—**
Bowman, F. C.

**UTAH**

**Eureka—**
Grandberg, Ericus

**Marysvale—**
MacCarthy, F. B.

**Midvale—**
Cullen, J. F.
Hamilton, E. H.

**Park City—**
Ellsworth, J. T.

**Payson—**
Moulton, Ernest

**Pleasant Grove—**
Hayes, J. J.

**Provo City—**
Smith, F. W.

**Salt Lake City—**
Cameron, F. K.
Fleming, E. P.
Koering, B. R.

**VERMONT**

**Northfield—**
Howard, S. F.

**VIRGINIA**

**Alexandria—**
Fawcett, L. H.

**Ivanhoe—**
Kearns, E. J.

**Norfolk—**
Webb, L. W.

**Parr—**
Davison, A. W.
Moxham, A. J.

**Roanoke—**
Brown, F. C., Jr.

**Saltville—**
MacMahon, J. H.

**University—**
Dunnington, F. P.

**WASHINGTON**

**Chewelah—**
Dolman, C. D.

**Hoquiam—**
Lamb, F. H.

**McCleary—**
Macke, J. C.

**Seattle—**
Casey, G. L.
Corey, C. R.
Gailey, W. R.
Magnusson, C. E.
Miller, A. A.
Peterson, J. C.
Ralston, O. C.
Rockwell, R. L.

**WASHINGTON—Continued**

**Seattle—Continued**
Ross, J. D.
Thomas, B.
Williams, C. E.

**Spokane—**
Armstrong, L. K
Keffer, Frederick
King, Rowland
Koeper, L. L.
Levy, G. J.

**Tacoma—**
Dunlap, T. E.
MacPherson, A. R.
Porro, T. J.
Zischkau, O. C.

**WEST VIRGINIA**

**Charleston—**
Pierce, J. B., Jr.
Pitcher, A. M.

**Clarksburg—**
Graves, C. M.

**Hartford—**
Strubing, F. W.

**Martinsburg—**
Kohlhausen, B. F.

**Morgantown—**
Clark, F. E.
Gregg, J. M., Jr.
Hite, B. H.

**Warwood—**
Bumgardner, J. W.

**WISCONSIN**

**Appleton—**
Phillips, C. A.
Wagner, H. A.

**Eau Claire—**
Thompson, J. G.

**Janesville—**
Stewart, C. O.

**Madison—**
Burgess, C. F.
Fleckenstein, C. F.
Helfrecht, A. J.
Kahlenberg, L.
Kowalke, O. L.
Meisekothen, E. E.
Ruhoff, O. E.
Schulte, W. B.
Storey, O. W.
Tan, Bo N.
Watts, O. P.

**Milwaukee—**
Adams, J. L., Jr.
Brown, C. G.
Chadwick, R. A.
Cochran, T. H.
Evans, C. T.
Kremers, J. G.
Raeth, F. C.
Wesle, E. H.
Zimmermann, J. G.

**Racine—**
Evans, D. G.
Waukesha, A.

**WYOMING**

**Casper—**
McKim, J. W.

**Thermopolis—**
Freeman, G. N.

## CANADA

### BRITISH COLUMBIA

**Anyox—**
Speight, H.

**Kimberly—**
Dahlgren, C. G.

**Nelson—**
Fowler, S. S.

**Trail—**
Beasley, F. E.
Lee, F. E.
Stimmel, B. A.
Willis, F. S.

**Vancouver—**
Barwick, W. S.
de Giovellina, E. C.
McIntosh, D.

### MANITOBA

**Winnipeg—**
Brown, G. J.

### NEW BRUNSWICK

**St. John—**
Vaughan, F. P.

### NEWFOUNDLAND

**Grand Falls—**
Judge, T. F.

**St. John's—**
Morris, J. W.

### NOVA SCOTIA

**New Glasgow—**
Cantley, T.
Chambers, A. R.

**North Sydney—**
Young, F. H.

### ONTARIO

**Belleville—**
Bennie, P. McN.

**Dryden—**
Mathews, F. P.

**Goderich—**
Kelleher, James

**Hamilton—**
Renton, W. L.

**Kakabeka Falls—**
Farrow, P. R.

**Kingston—**
Cadenhead, A. F. G.
Goodwin, L. F.

**London—**
Sturdevant, E. G.

**Merrickville—**
McMullen, G. P.

**Merritton—**
Hedalen, John

**Niagara Falls—**
Arison, W. H.
Doyle, H. L.
French, E. L.
Gardner, G. N.
Offerhans, W. R.

### ONTARIO—Continued

**Nickelton—**
Gillis, J. H.

**Ojibway—**
Baltzell, W. H.

**Orangeville—**
Deagle, L. M.

**Orillia—**
Lamble, B. C.

**Ottawa—**
Schjelderup, G.
Simpson, Louis

**Renfrew—**
Cram, W. C., Jr.

**Sandwich—**
Walker, G. H. P.

**St. Catharines—**
Sieger, G. N.

**Sudbury—**
Holst, G.

**Thorold—**
Schabacker, H. E.

**Toronto—**
Allan, W. G.
Bain, J. W.
Barrows, W. S.
Benson, G. C.
Brown, N. B.
Burpee, L.
Burt-Gerrans, J. T.
Chisholm, T. H.
Clark, F. C.
Gaby, F. A.
Gifford, W. M.
Guess, G. A.
Hedstrom, E. S.
Kendrick, F. B.
Lytle, L. B.
MacDougall, A. J.
Miller, W. L.
Moffat, J. W.
Robertson, F. D. S.
Roumbanis, D. C.
Shepherd, F. A.
Wood, E. M.

**Welland—**
Young, John

### QUEBEC

**Buckingham—**
Hambly, F. J.

**Deschenes—**
Strom, Birger
Torell, T. F.
Waddell, H. R.
Westby, G. C.

**Hull—**
Wilson, J. R.

**Joliette—**
Disario, John

**Montreal—**
Crafts, W. N.
Davidson, T. R.
Eardley-Wilmot, T.
Falter, P. H.
Gilman, M. C.
Kaelin, F. T.

## CANADA—Continued

### QUEBEC—Continued

**Montreal—Continued**
Lindstrom, R. L.
Matheson, H. W.
Morgan, N. L.
Sissener, John
Stansfield, A.
Wales, J. A.
Woodyatt, J. B.

### QUEBEC—Continued

**Shawinigan Falls—**
Acton, E. H.
Crowther, C. W. W.
Kelly, J. J.
Williams, E. R.
Witherspoon, R. A.

**St. Lambert—**
Platts, J. C.

## CUBA

**Guantanamo—**
Simon, E. J.

**Havana**
De Milan, Alex
Hill, R. H.
Smith, J. M.

## HAWAII

**Honolulu—**
Castle, S. N.

## MEXICO

**Coahuila—**
Shaw, S. F.

**Hidalgo—**
Kuryla, M. H.

**Mexico City—**
DeLandero, C. F.
Hinton, G. B.
Rost, H. F.
Santillan, R. A.

**Monterrey—**
Berthier, U. H.

**Orizaba—**
Wilkins, E. M.

**Truinp—**
Stevens, B.

## PANAMA

**Gatun—**
Grier B. R.

## PHILIPPINES

**Manila**
Herman, H.
Zamora, M.

## PORTO RICO

**Insenada—**
Boyd, T. D., Jr.

## SOUTH AMERICA

### BOLIVIA

**Oruro—**
Ferron, R. D.

### BRAZIL

**Rio de Janeiro—**
De Medeiros, T. S. V.
Maya, P. de C.

**S. Paulo—**
Bowles, R. H.
Byington, A. J.
De Souza, E.
Pompeia, Jonas

**Villa Nova de Lima—**
Jones, H.
Wilder, F. L.

### CHILE

**Antofagasta—**
Rowe, G. M. W.

**Chuquicamata—**
Carlton, A. C.
McClenahan, J. S.

### CHILE—Continued

**Iquique—**
Boesch, J. E.
Lipscomb, G. J.

**Santiago—**
Cardoen, Remy
Carr, H. D.
Diaz-Ossa, B.
Hayward, R. F.

### PERU

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Rosenshine, L. J.

**Cerro de Pasco—**
Gahl, Rudolf

**Oroya—**
Dalmeida, J. A.
Mather, T. W.

### VENEZUELA

**Caracus—**
Dalburg, F. A.

## AUSTRALIA

### NEW SOUTH WALES

**Port Kembla—**
White, E. A.

**Sydney—**
Murphy, R. K.
Nicholson, J. B.

**Wallaroo—**
Hancock, H. L.

**Yetholme—**
Du Faur, J. B.

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**Auckland—**
Crookes, S. T.

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Henderson, J. B.
Jackson, A. G.
Just, J. S.

**Mt. Morgan—**
Johns, M. J.

**Woodend Ipswich—**
Lawrenson, J. S.

### TASMANIA

**Hobart—**
Butters, J. G.

**Queenstown—**
Sticht, R. C.

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Chappell, W. C.
Clements, F. W.
Gepp, H.W.
Klug, G. C.
Leggo, A. V.
Wood, B.

## ASIA

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**Chunking—**
Loh, F. C.

**Hankow—**
Wang, C. Y.

**Hanyang—**
Woo, Z. T. K.

**Hong Kong—**
Lee, L. P.
Wong, K. T.

**Lincheng—**
Kwang, K. Y.

**Shanghai—**
Chow, Jen
Gaither, R. H.
Zen, H. C.

**Sinti City—**
Leo, S. T.

**Szechuen—**
Chiang, Y. K.

**Tayeh—**
Wong, W. A.

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Srinivasayengar, V. K.

**Bombay—**
Maneckji, J. B.
Vakil, K. H.

**Calcutta—**
Eldredge, M.

**Madras—**
Green, L. B.

### JAPAN

**Akita—**
Ikeda, K.
Satoh, H.

**Chickuzen—**
Yasukawa, D.

**Ehimeken—**
Yabe, C.

### JAPAN—Continued

**Fukuoka—**
Araki, A.
Higashi, S.
Kaneko, K.
Tatsumi, E.

**Fukushimaken—**
Kitsunezaki, I.
Nakashima, Shigemaro

**Funatsu Hida—**
Sato Hisao

**Fushimi—**
Tonamy, C. H.

**Gifuken—**
Kumami, A.

**Heyogoken—**
Abe, Asaka

**Hyogo-ken—**
Murahashi, S.

**Ishikawaken—**
Iwai, Kyosuke

**Komatsu—**
Takikawa, Y.
Yano, M.

**Kawasaki—**
Matsushita, N.
Shinjo, Y.

**Kobe—**
Isobe, F.
Tachihara, Jin
Tada, S.

**Kogoshima—**
Maruyama, J.

**Kumamoto—**
Taniguchi, K.

**Kyoto—**
Nakasawa, Yoshio
Namba, M.
Nishikawa, Kikei
Takenouchi, I.
Yoshikawa, K.

**Nagasaki—**
Nakamura, M.

## ASIA—Continued

### JAPAN—Continued

**Nogano—**
Takahashi, Taketare

**Nagoya—**
Mine, S.

**Nikko—**
Kaku, Juroku

**Osaka—**
Arakawa, E.
Asano, K.
Itoh, Ichiro
Kawakita, Y.
Kimura, Z.
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Nakas, K.
Takakuwa, K.
Tsujimoto, K.
Yamamoto, I.
Yunoki, S.

**Sendai—**
Fuseya, G.

**Taiwan—**
Jinguji, G.

**Tokyo—**
Fumoto, S.
Gotoh, Issaku
Horibe, Y.
Igarashi, T.

### JAPAN—Continued

**Tokyo—Continued**
Incuye, T.
Ishikawa, Ichiro
Ito, Ichiro
Kai, Hideo
Kameyama, Naoto
Kato, Y.
Katsura, B.
Kawamura, T.
Kishi, K.
Kitawaki, I.
Kurahashi, T.
Nakahara, S.
Nishida, Hirotaro
Nishioka, S.
Nohara, T.
Ohashi, F.
Oshima, Y.
Shigematsu, Y.
Tabata, K.
Tanaka, J.
Watanabe, H.
Yamaguchi, K.
Yamasaki, J.

**Wakayama-ken**
Tada, Y.

**Yokohama—**
Tada, K.

## AFRICA

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Haas, S. W.

**Broken Hill—**
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Schwartz, Hyam

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Deakin, Gerald

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Solvay, A.

**Couillet—**
Hutin, E.

**Jemeppe—**
Queneau, A. L. J.

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Henault, O-Dony.

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Raeder, Bjorn

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**Abo—**
Aminoff, G.

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Caudrelier, E.
Champin, A. P.
deGeofroy, A.
DeSallier, Rene
Gall, H.
Halter, Georges
Hansson, A. S.
Keller, C. A.
Marie, C.

### FRANCE—Continued

**Paris—Continued**
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Roberts, W. S.

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Humbert, E. P.

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Brander, J.

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Winkley, S. F.

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**Dologarrog (Wales)—**
Paton, G. K.

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Scott-Maxwell, J. M.

**Hatch End—**
Jacob, Arthur

**Irvine (Scotland)—**
Walker, J. W.

**Leeds—**
Salter, Chas.

**Lincoln—**
Freeston, T. A.

## EUROPE—Continued

### GREAT BRITAIN—Continued

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Brown, P. Hunter
Carnegie, E.
Chalas, A.
Frink, R. L.
Goldstein, P.
Lewis, G. P.
Lloyd, G. C.
MacKness, C. F.
Marshall, A. L.
Merz, C. H.
Rondelli, T.
Sprague, E. C.
Shields, John
Smith, R. T.
Walters, George

**Newcastle-on-Tyne—**
Rollin, Hugh

**Norton-on-Tees—**
Johnson, A.

**Sheffield—**
Cutts, V. O.
Etchells, H. E.
Fawcett, P.

**St. Helens—**
Harrison, A.

**Sunbury-on-Thames—**
Cowper-Coles, S.

**Tunbridge Wells—**
Colquhoun, James

**Walthamstow—**
Wilder, F. L.

**Widner—**
Pritchard, D. A.

**Yiewsley—**
Buckle, H. L.

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Eknes, L.

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Cambi, L.
Merizzi, G.
Paravicini, R.
Rossi, C.
Toniolo, Carlo

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Chiaraviglio, D.

**Touno—**
Giolitti, F.

**Trentino—**
Wright, C. A.

**Turin—**
Gardiol, A. D.

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**Delft—**
Reinders, Wm.

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### NORWAY

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Roll-Hansen, C.
Turner, M. R.
Tysland, Geo.

**Borgestad—**
Knudsen, Rolf

**Christiania—**
Baararnd, K.
Berg-Hansen, B.
Bjornson, E.
Brodal, P.
Collett, E.
Collett, Ove
Dedichen, Herman
Farup, P.
Goldschmidt, Heinrich
Hiorth, A.
Hiorth, F. V. L.
Homan, C. H.
Johansen, G. H.
Krog, K. M.
Lenschow, H. H.
Ogle, H. L.
Olsen, T. S.
Raeder, B.
Rafu, Wm.
Saeter, H. B.
Sem, Mathias
Somdal, J. A.
Stephanus, A. D.
Tharaldsen, Filip
Thorne, C. A.
Von Krogh, Johan

**Christiansand—**
Frisak, Arvid
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Hole, Ivan
Westly, J.

**Drammen—**
Sturbelle, L. C.

**Fredriksstad—**
Moltkehansen, I. J.
Washburn, W. F.

**Hoyanger—**
Murer, Johan

**Nakkerud—**
Loken, R.

**Odda—**
Andersen, Jorgen
Giersten, S.
Gjerding, E.
Johnson, E. B.

**Porsgrund—**
Kaufmann, E.

**Rjukan—**
Bonnevie, H.
Nilssen, Bjarne

**Ryfylke—**
Horn, H. T.
Sneath, W. H.

**Stavanger—**
Edgeberg, B.

**Trondhjem—**
Pedersen, H. C.
Simonsen, I. B.

## EUROPE—Continued

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Billings, A. W. K.
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Lana, C.

**Beinosa—**
Lehmskuhl, C. U.

**Reus—**
Miro, A. R.

**Tarrasa—**
Balta de Cela, Jose

### SWEDEN

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Berglund, E. S.

**Falun—**
Svensson, C.

**Gullspang—**
Hedlund, M.

**Hagge—**
Yngstrom, L.

**Orebro—**
Broms, H.

**Sodertelje—**
Tesch, H. G. A.

**Stockholm—**
Carlson, Birger
Demmler, A. W.
Gronwall, A.
Harden, J.
Kring, Oskar
Leffler, J. A.
Lilja, S. G.

### SWEDEN—Continued

**Stockholm—Continued**
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Martin, Herbert
Olsson, H.
Palen, A. G. P.
Palmaer, K. W.
Wahlberg, A. F.

**Stocksund—**
Liljenroth, F. G.

**Stromstad—**
Halldin, C. H.

**Trollhattan—**
Andersen, F.
Forssell, J.

**Uddeholm—**
Herlenius, A.

**Vesteras—**
Edstrom, J. S.

**Westervik—**
Tillberg, E. W.

### SWITZERLAND

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Engi, Gadient
Fichter-Bernoulli, F.

**Geneva—**
Guye, P. A.
Lacroix, H.

**Turgi—**
Landolt, H.

**Zurich—**
Wunderly, C.

*A paper presented at the Thirty-eighth General Meeting of the American Electrochemical Society, in Cleveland, October 1, 1920, President Landis in the Chair.*

## FARADAY'S LAW AT THE CATHODE.[1]

By J. W. Richards.[2]

D. A. LYON

Careful consideration of the phenomena of electrolysis, particularly much experimental study of the relation of the anion and cation to the current passing, has raised in my mind the doubt as to whether the cation can be said to be directly amenable to Faraday's law. In other words, to put the question bluntly: Is the amount of cation liberated directly dependent upon a proportional amount of current passing, or is it secondarily or indirectly dependent thereon, being simply that amount of the cation which is chemically combined in the electrolyte with the amount of anion being liberated? In ordinary chemistry, in discussing various compounds of a metal with different quantities of an acid element, such as oxygen, chlorine, etc., we take it for granted that the acid element has constant valence while the valence of the metal varies in its different compounds. This is perhaps not a mere assumption, but is forced upon the theoretical chemist by many considerations, so many that it is at the present time taken for granted, as a matter of fact. This would point to the invariability of the valence of the anodic element and therefore of a constant relation between its quantity and the electric charge it carries; on the other hand, it would postulate a varying valence of the cathode element, that is to say a varying or indirect relation between the quantity of that element and the cathodic electric charge.

Whatever the real chemical reasons are, it is a matter of fact that the chemist speaks of stannous and stannic salts, mercurous and mercuric salts, cuprous and cupric salts, etc., etc., in all of which the invariability of the valence of the acid constituent is assumed and the variability of the valence of the basic element

[1] Manuscript received August 9, 1920.

[2] Professor of Metallurgy, Lehigh University.

postulated. In electrolysis, the conventional direction of the current is from the anode to the cathode, but modern views incline to assuming the real direction of the current to be the opposite of this, *viz.*, that streams of so-called negative electrons pass from the cathode to the anode. The existence of positive electrons, travelling in the opposite direction, is not established, and the tendency is to substitute for this conception the idea of a deficit of negative electrons, and thus complete a unitary theory of the electric current.

Since the acid element of a compound being electrolyzed is liberated at the anode, we can at once assume a direct relation between the amount of acid element set free and the quantity of negative electrons traveling towards the anode, in other words, that 96,500 coulombs, or one Faraday, accompanies one valence weight of acid element set free at the anode, or corresponds to one valence of chemical work done.

At the cathode, the amount of cathode element set free while one Faraday of electricity passes is a variable, according to the assumed variable valence of the cathodic element. It may be one Faraday is equivalent to a half, a third or a fourth of an atom weight, corresponding to the (assumed) chemical valency of the cathodic element.

It stands therefore, as a matter of fact, and not at all a question of theory, that the relation between the current passing and the quantity of chemical element liberated at the anode is invariable and direct, while the relation between the electric current passing and the amount of cathodic element concerned is a variable, and therefore probably indirect.

The thesis which the writer wishes to bring forward for discussion is therefore the following:

Since the direct invariable relation between electrical current and chemical quantity subsists only at the anode, and the present theory of electric current postulates current passing, as negative electrons, only towards the anode, is it not logical and reasonable to assume that the direct relation between electrical current and chemical quantity is true only at the anode?

On the other hand, the relation at the cathode being variable, and therefore probably indirect, is it not logical and reasonable to assume that this relation is not electrochemical but is more

properly designated as chemical only; that is, it is governed by the chemical relation between the quantity of anodic element set free at the anode and the cathodic element. In other words, when the electric current sets free electrochemically at the anode a given amount of the anodic element or constituent of the compound, an amount directly determined by the electrochemical relations and Faraday's law, there is set free at the cathode that amount of the cathodic constituent which was chemically combined with the amount of anodic constituent liberated. This relation is chemical, and determined by the chemical equivalents of the substances in question, and subject to the variable valence of the cathodic element just as in any simple chemical decomposition.

If the above reasoning is valid and logical, it would follow that Faraday's law will apply fundamentally only to the anodic constituent, which is directly related to the quantity of negative electrons traveling to the anode; it would correspondingly follow that Faraday's law does not directly apply to cathodic constituents, but only indirectly through the chemical relation that the quantity of cathodic element liberated is simply that which was chemically combined with the quantity of anodic constituent liberated, and is only thus indirectly related to the quantity of electric current passing.

*Metallurgical Laboratory,*
*Lehigh University.*
*August 7, 1920.*

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## DISCUSSION.

CARL HERING[1]: The same point that Professor Richards has raised has come up before, and I myself have called attention to the fact that Faraday's Law was stated incorrectly in most books. In some cases it does not work out right, but if you change the word "valence" in Faraday's Law to "change of valence," it

[1] Consulting Electrical Engineer, Philadelphia, Pa.

covers every case and then it applies to the cathode as well as to the anode.

A MEMBER: Will you please state Faraday's law?

CARL HERING: I would not venture to give an off-hand definition of it now, but the words "change of valence" are not generally stated in Faraday's Law in the books; if you make this change in the law it will apply to all cases, including those in which you do get twice as much copper per ampere-hour with one solution as with another.

W. S. LANDIS[2]: I think a paper of this kind presents a rather interesting matter of policy to this Society. I know I have looked up Faraday's Law many a time, and it depends on the book you read as to what Faraday's Law is, or rather the way it is stated. It seems to me that it would be well to precede this paper with an exact statement of Faraday's Law, so we younger men would have something to tie to. I can take half-a-dozen books of physics and chemistry, and every one has a different statement of Faraday's Law, probably intended to mean the same thing, but they are not in the same language.

CARL HERING: I suggest that Professor Richards add this.

J. W. RICHARDS: I would state the law under discussion as follows: The electrochemical actions produced at the electrodes by given currents are proportional to the chemical equivalent weights of the bodies affected by the current.

C. P. MADSEN[3]: I would like to ask the author if he knows of any specific instance where it would make any difference in practical results over those obtained by Faraday's Law?

J. W. RICHARDS: I do not know of any case where it would make any actual difference in the experimental facts. The only difference is in our conception of what is happening. It seems to me there is a difference in our mental attitude as to whether or not we consider that the electric current, according to the old view, is passing in two directions, that is, liberates at the anode certain amounts of anion in proportion to the current passing, and at the cathode, certain amounts of cation in propor-

[2] American Cyanamid Co., New York City.
[3] Consulting Engineer, New York City.

tion to the current passing, both having a direct relation to the amount of current passing; that is one conception. The other is that the relation between the chemical action and the electricity passing is direct only for the anion, and that the amount of cation liberated is not controlled directly by the amount of electricity passing, but is simply that amount of cation which was combined with the anion which was liberated. It is simply a difference in point of view; there is no difference in the experimental facts, but it is a question which way you are going to look at it. I think that Dr. Hering's statement is the best foundation to stand on; that is, putting it in modern language, that the passage of 96,494 coulombs makes a change of valence of one in a gram equivalent of material affected. That will apply at both anode and cathode, and perhaps give some answer to the question raised here.

CARL HERING: May I add that this way of interpreting it sometimes involves negative and zero valences, which some chemists do not like.

J. W. RICHARDS: In which I, speaking as a chemist, thoroughly agree with Dr. Hering's point of view.

WM. BLUM[4]: Just one point Dr. Richards mentioned might be misleading; I do not think he intended it that way. He spoke of only the cations as having variable valence. Of course we must recognize that the elements in the anions may undergo a change of valence, *i. e.*, we can have changes of valence occurring both at anode and cathode. At the anode, however, this does not occur without changing the composition of the ion.

J. W. RICHARDS: Do you know of any case where an anion has a variable valence in combinations? I assume, of course, that when liberated, as free element, its valence is zero.

WM. BLUM: No, and one reason is that we define the valence of the elements in terms of the anions, on the assumption that the valences of the anions remain constant.

J. W. RICHARDS: Exactly, and it is a curious fact that the chemists have virtually made that tacit assumption without questioning it.

[4] Chemist, Bureau of Standards, Washington, D. C.

*A paper presented at the Thirty-eighth General Meeting of the American Electrochemical Society, in Cleveland, October 1, 1920, President Landis in the Chair.*

# A NEW FORM OF STANDARD CELL.[1]

By C. J. Rodman[2] and Thos. Spooner.[3]

## Abstract.

[A new portable standard cell which is a satisfactory laboratory standard has been developed. It is a cadmium cell with the usual ingredients but having a container of hard glass with tungsten leads. Instead of the common H-type a compact concentric arrangement has been devised possessing a number of advantages. This cell is made either saturated or unsaturated, and other combinations besides the standard cadmium formula may be used in the present cell blank if desired. Methods of purifying the chemicals and filling the cell are dsecribed.]

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## INTRODUCTION.

A dependable, compact, portable standard cell has been greatly desired and it was with the idea of improving these factors that the development of a new form was undertaken. Much has been written on the subject of standard cells, and undoubtedly the best combination for great stability is the cadmium cell. The chemicals, Hg | $Hg_2SO_4$ | $CdSO_4$ (saturated) | Cd amalgam, which enter into its composition may be easily prepared in a pure state, if proper precautions are adopted. Accordingly the cadmium type only has been seriously considered.

In order to increase the dependability, new materials for the container and leads have been tried and adopted. In order to increase the compactness, the old H-type of cell has been abandoned and a concentric type having a diameter of about 1 in. (2.54 cm.) and a height of about 3.5 in. (8.9 cm.) has been developed.

[1] Manuscript received July 28, 1920.
[2] Research Chemist, Westinghouse Electric & Mfg. Co.
[3] Electrical Engineer.

## DEVELOPMENT OF CELL.

*New Materials.* The high percentage of failures of cells of soda glass with platinum leads, of the H-type, led us to believe that this condition could be altered by selecting another combination of metal and glass. Soda glass has a coefficient of expansion of $8.33 \times 10^{-6}$, and platinum $8.99 \times 10^{-6}$ at room temperatures, so that even well annealed seals often cause leaks or cracks in the glass. Other factors, as mechanical strength and chemical stability of soft glass, failure of seals due to zinc amalgam of the Clark cell causing the platinum contact wires to crack the glass, etc., may be cited as reasons why other combinations have been tried. It is obvious that as leading-in wires only metals can be used which will be inert to any constituent that the finished cell contains.

The latest design consists of a hard silica glass container which has a coefficient of expansion of $3.50 \times 10^{-6}$, and tungsten leads of $3.60 \times 10^{-6}$. In selecting a stable hard glass for this purpose we have chosen a high silica pyrex. Such glass has been found to be highly insoluble toward the slightly acidic properties such as we encountered in standard cell conditions. In studying the osmotic electric E. M. F., Carmen and Fritts found pyrex to be very superior to Jena glass for similar purposes. As shown in their summary, pyrex proved to be approximately three times as insoluble as Jena glass. Such compositions as sulphur, rosin, sealing wax, etc., make quite an appreciable difference in cell construction. Some of the latter ingredients have been used in certain makes of standard cells for plugs and seals and have shown no drastic ill effects, but where the best equilibria are desired it is advisable not to use them.

In order to make a good tungsten-glass seal it is necessary to thoroughly clean the metal beforehand. Boric acid is used for this purpose. A thin coat of $B_2O_3$ is allowed to remain upon the wire after the fluxing action has taken place. It is necessary to heat the tungsten to a cherry red during the sealing-in of the wire. Most of the thin coat of $B_2O_3$ is taken up into the glass. Should any be left free, upon the wire, it should be completely washed off with water when the cell is cleaned. $B_2O_3$, as well as other acid oxides occurring in the glass, when taken up by the cadmium sulphate solution have a tendency to depress the E. M. F. of the

cell, whereas other oxides in the solid solution of the glass such as PbO, ZnO, $Na_2O$, $K_2O$, etc., when dissolved in the solution increase the E. M. F. Alumina and silica as such have no noticeable effect upon the cell equilibria. However, with the glass used no appreciable effect was observed due to the interaction of the glass with the cell ingredients.

It is interesting to note that the cell contents penetrate the high lead and basic oxide glasses to an appreciable depth depending upon the time of standing of the cell. By emptying the cell and heating the blank to the temperature of the softening point of the glass and cooling, a definite crazing effect may be observed, that penetrates uniformly to a depth of a few tenths of a millimeter. Similar phenomena are produced by acid treatment and heating of certain glasses.

The cells made of hard glass with tungsten seals are very strong and durable. No failure of the many cells made thus far can be attributed to the hard glass-tungsten junction, whereas many of the soft glass cells with platinum leads have proven mechanically weak, have crazed or cracked. A few of the H-type cells were constructed of 702-P glass with tungsten leads. When they were carefully filled and kept in an upright position such early forms made very good laboratory standards.

## *New Forms.*

For constant laboratory use stable portable cells are needed. The first attempt to produce such a cell resulted in the modification of the H-type. The lower limbs were somewhat enlarged spherically, being about 1.5 cm. in diameter and restricted to a tubular opening about 0.5 cm. for the remaining part of the legs. The cross-arm was enlarged at the center to about 2 cm. in diameter and tapered to 0.5 cm. where it joined the legs. At the joints small circular porous porcelain diaphragms of the Hulett voltameter type were sealed in and separated the cell into three compartments. The lower sphere of one limb was filled half full of mercury, upon which was packed mercurous sulphate paste. The mercurous sulphate-mercury contact surface was at the widest place in the sphere. The paste filled the leg to 0.5 cm. in depth. Above the spherical portion small clear crystals of $CdSO_4.8/3H_2O$ filled the remaining portion of the compartment, thus holding the

leg constituents in place no matter in which position the cell was orientated. The amalgam of the other leg, being frozen, easily holds itself in position at room temperatures. Saturated cadmium sulphate solution completely filled the central chamber and the intervening space between the crystals of both limbs. A satisfactory cell was thus made, but its construction was somewhat difficult due to the sealing-in of the diaphragms. This form was later abandoned in favor of the concentric type.

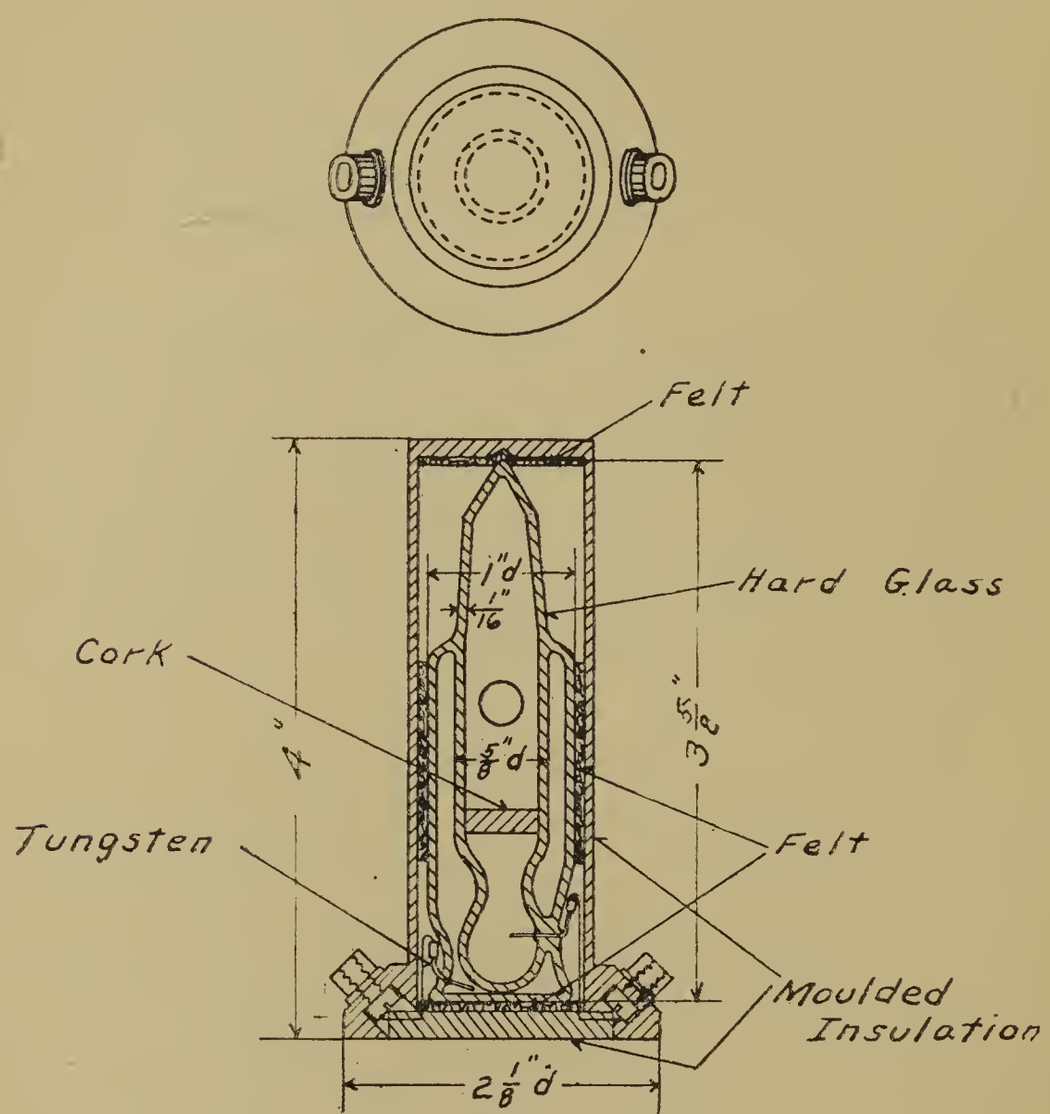



Fig. 1.

In Fig. 1 a diagram of the concentric type of standard cell is shown drawn to scale. The outside dimensions of the cell blank are 2.5 cm. diameter x 10.0 cm. high. The outer compartment is tubular in form with closed bottom, and is slightly indented near the lower end so that the tungsten leading-out wires can be bent sharply at right angles just outside the glass wall, thus leaving a form which is flush with the diameter of the outer

compartment. A glass tube 1.6 cm. outside diameter is sealed concentrically with the outside glass tube and forms the cathode leg of the cell. In addition to being sealed at the upper constricted portion of the central tube, which is about 6 cm. from the bottom of the cell, it is sealed at a point near the bottom and furnishes contact with the outer glass tube so that a tungsten lead can be made to connect the inner portion of the cathode without being electrically connected with the anode chamber. Just below the upper seal and about 5 cm. from the bottom of the cathode, a hole about 1 cm. in diameter serves to connect the anode chamber, which can be filled from that point. The cathode tube extends several centimeters beyond the upper joint so that after the cell is filled it can be conveniently drawn out and sealed off. Tungsten wire No. 24 being very stiff and difficult to bend when cold without breaking, led to the construction of a lead which fulfills the purpose very well.

Just outside the tungsten leading-in wire, a copper wire was brazed to the tungsten which served the purpose, provided the copper wire was not twisted or jerked. However, in cells which are being used in the laboratory, in which the seals are not rigidly protected, the copper wire in this case had a tendency to break off near the weak bronze joint. Moreover, great care had to be taken not to heat the copper too hot while brazing because of the high $Cu_2O$ formation within the wire causing high resistance and subsequent breaks. To remedy this difficulty a tungsten-monel-copper joint was made by electro-spot welding. Such a combination holds very tenaciously and if the copper wire is redoubled back a short distance on the tungsten and wound about the joint a very flexible lead is made. The difference in temperature between the leads on either side of the cell is very small. The correction for the thermal E. M. F. is less than $2.5 \times 10^{-7}$ volts per degree. The contact E. M. F. in the leads is obviously balanced one against the other as the finishing lead consists of copper wire. The cells are mounted in a bakelite-micarta case fitted with suitable binding posts. The top, bottom and sides of the cell proper are protected from breakage by felt pads which will absorb any but very severe shocks to which the cell might be subjected. Although the diameter of the cell base is only about 2 inches (5 cm.) the cell is very stable, since the center of gravity is low due to

the mercury near the bottom. Figs. 2 and 3 show by comparison the composition of the new form of cell. Due to the low thermal conductivity of the walls of the container and the high thermal conductivity and intimate contact of the two legs of the cell proper, external changes of temperature should have a small effect on the E. M. F. of the unsaturated cell.

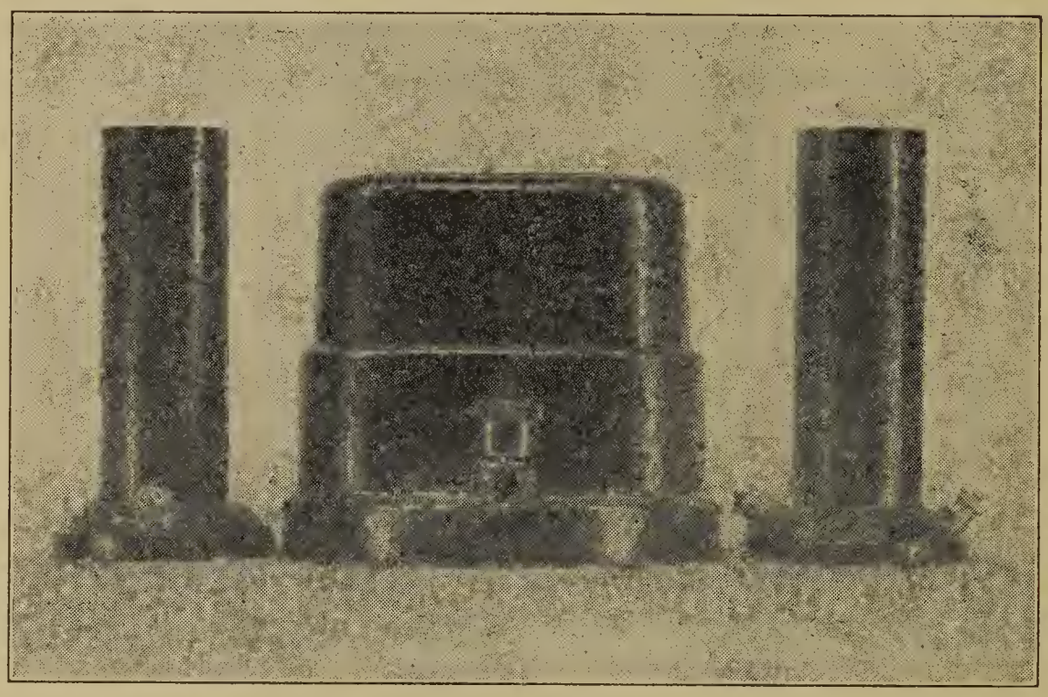


Fig. 2.

PURIFICATION OF THE CELL INGREDIENTS.

*Chemically pure mercury* obtained on the market usually contains many metals and must be specially purified before using it in standard cells. Many methods have been devised from time to time for this purpose, yet few are comparatively rapid while thorough in purification. The following method has been used with success in producing pure mercury from the technical supply and even from waste amalgam caught in the trap of a sink.

Fig. 4 shows diagrammatically the scheme used in purification. From 2 to 8 kilograms of impure mercury are introduced into the large container *V*. Upon rapid stirring, about 1500 R. P. M., the mercury is drawn up through the tube at the bottom of the stirrer *A* and thrown out through the arms, thus breaking the mercury into a great number of fine globules. At a speed of 2500 R. P. M., the globules of mercury are piled up from one

to 2 cm. in depth above the mercury proper. A great deal of surface is thus exposed to the action of dilute nitric acid (3 per-cent), which filled the vessel *V* above the opening to the cell *E*. The small auxiliary cell, which is about 1.5 cm. in diameter, contains a platinum electrode 3 x 6 cm. square, which is made cathode when applying a six-volt drop between the leads *M* and *N*. The cathode current density was kept about 10 amperes per square decimeter, whereas the anode C.D. is comparatively low. By continuous acid washings and electrolysis the metals higher in the electromotive series are gradually deposited with mercury on the cathode. The resulting amalgam frees itself from time to

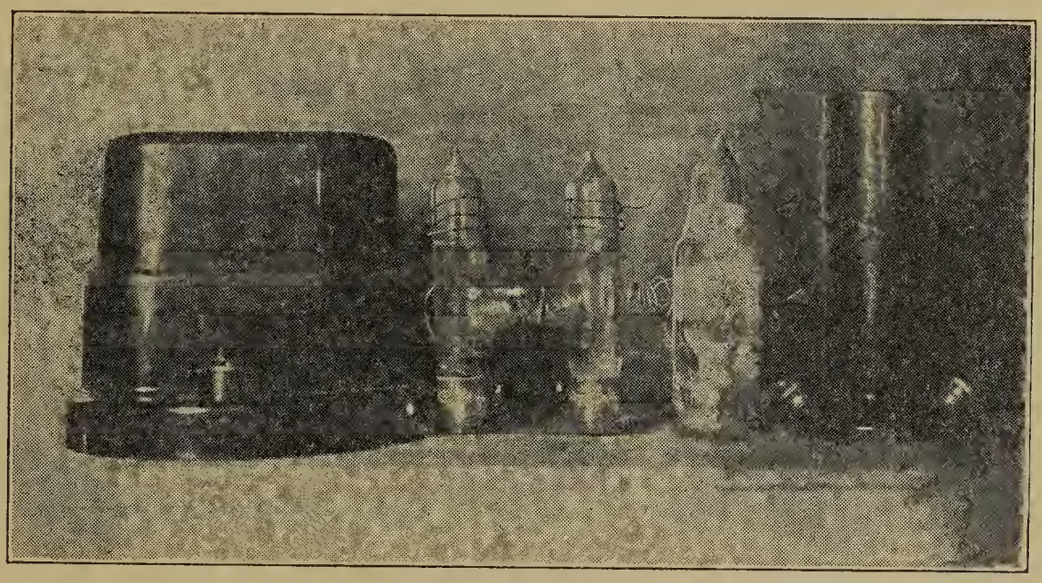


Fig. 3.

time and drops down to the bottom of the cell and empties through the small siphon at the bottom of the auxiliary cell into the waste. A comparatively small amount of mercury is deposited as waste. After continuing the process for a few hours, the mercury is drained off through the stopcock *K* and distilled in the vacuum still. If mercury of extreme purity is not desired the latter operation may be dispensed with.

The stirrer *A* is made of hard glass and sealed to a molybdenum rod 3/32 in. (2.35 mm.) in diameter. The rod is held by the set screw in the rotating apparatus which is driven by a small fan motor. The metal glass joint has withstood the vibration accompanying stirring as high as 2500 R. P. M.

*The mercurous sulphate* was prepared by the electrolytic method. The apparatus used, Fig. 5, is a unit of the Westinghouse electro-analysis bench, which has an unusually wide lati-

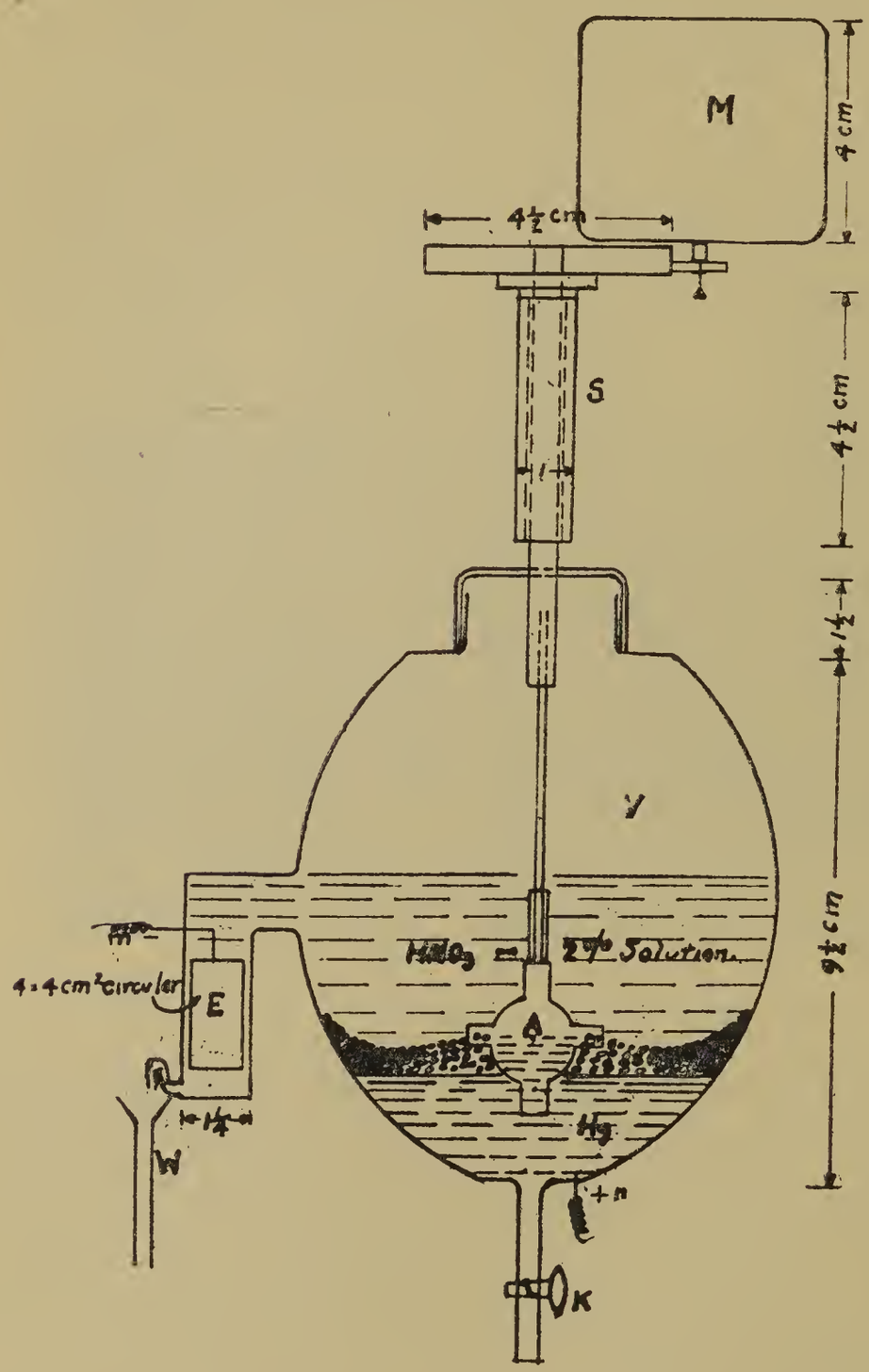



FIG. 4.

tude of regulation. Mercury, as purified above, is placed in a 600 cc. glass beaker to a depth of about 4 cm. 4 N. $H_2SO_4$, C.P., fills the beaker to within a few cm. of the top. A platinum lead through the bottom of the beaker in the mercury is made the

anode, whereas the small platinum gauze electrode near the top of the acid electrolyte is the cathode. Electrolysis is carried on with about 4 amperes per square decimeter anode surface. The hard glass stirrer turning at 350 R. P. M. keeps the solution well agitated and gives a fresh mercury surface continuously. The formation of large globules of mercury has no detrimental packing effect upon the sulphate, as these rapidly sink into the mercury and assist in keeping a fresh anode surface. Some fine globules are mixed with the sulphate and assist in making a good, clear, mercurous product. The vigorous stirring keeps much of the mercurous sulphate in suspension.

After a sufficient amount of product has been formed, it is transferred to a Gooch crucible and drained of excess electrolyte and of mercury by slight pressure upon the surface of the mass. Several washings with very dilute sulphuric acid, followed by rapid washings with anhydrous acetone or absolute alcohol, then with dilute cadmium sulphate, and finally with saturated cadmium sulphate solution, gives a good uniform product ready to make the paste.

*The paste* is made by using mercurous sulphate as prepared, mixing it with about 10 percent by volume of about 50-mesh (0.5 mm.) $CdSO_4.8/3H_2O$ crystals, and making it of the consistency of thick cream by the addition of saturated $CdSO_4$ solution.

*Cadmium* obtained from J. T. Baker was first redistilled in quartz in vacuum and was deposited in pure crystalline form. In freeing the crystals from the quartz surface when cold, small particles of quartz were chipped off with the crystals, so that this process was abandoned in favor of distillation in hard glass in vacuum. The product obtained by means of hard glass distillation was very pure, and could be freed from the receiver best by breaking the glass. Amalgam consisting of 12.5 percent by weight of cadmium was made by dissolving the purified cadmium in mercury in a covered weighing bottle placed upon a hot plate slightly above 100° C. The amalgam could be kept easily in this state until ready for use.

*The cadmium sulphate* as received from Baker and Adamson Co. contained small amounts of iron, nickel, zinc, and traces of other impurities, so that it was necessary to resort to further

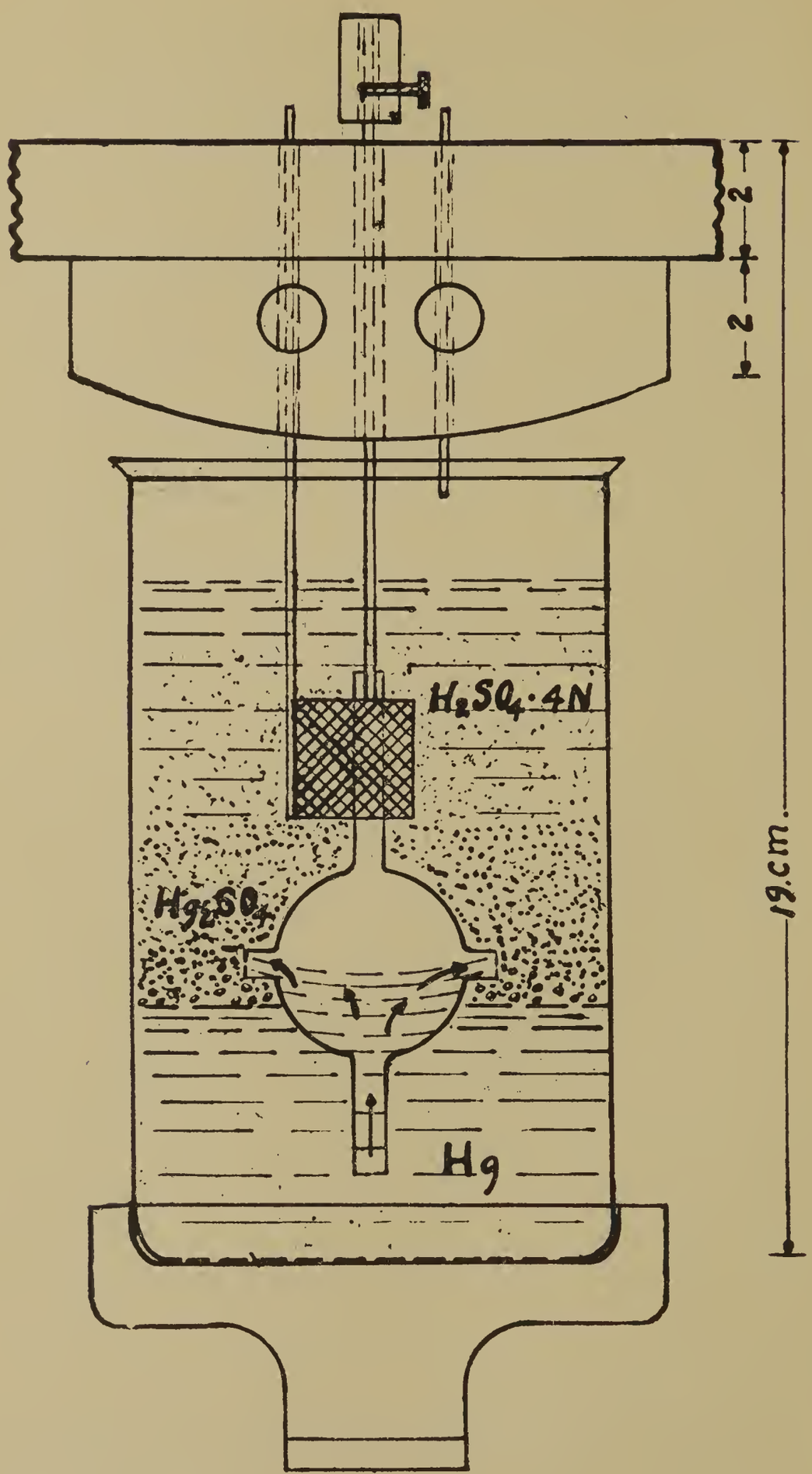



FIG. 5.

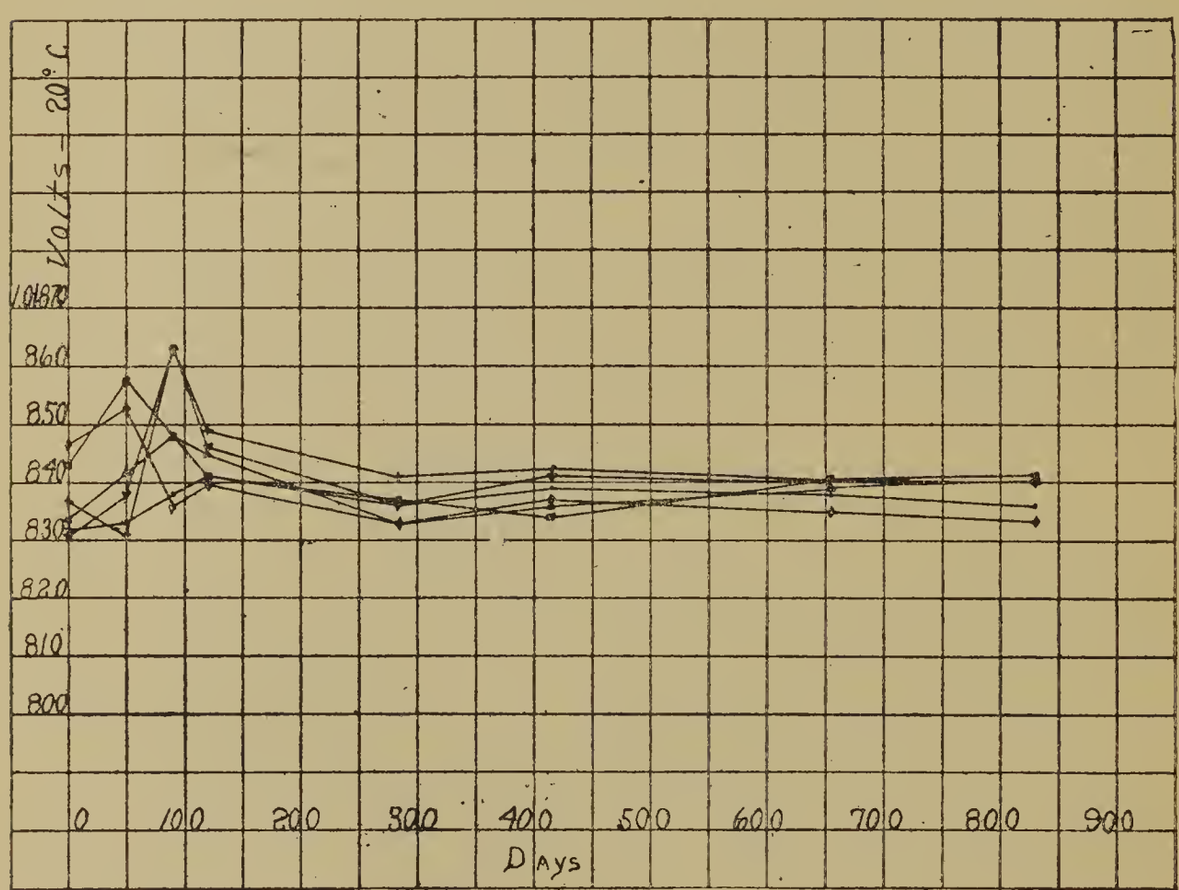
purification of this material. The crystals were dissolved in an excess of water during rapid agitation of the solution and crystals. A small amount of cadmium oxide and hydrogen peroxide were added to the solution, which then was placed in a low temperature (60° C.) oven and kept there for a few hours. After filtration of the ferric hydroxide and other precipitated material, a few drops of pure concentrated sulphuric acid were added. Upon continued heating the cadmium sulphate crystallized out and was collected by draining upon a Buchner funnel. Rinsing with a little distilled water and drying gave a quantity of cloudy crystals. These were dissolved in water giving a nearly saturated solution. A few drops of $H_2SO_4$ were added and the solution was set aside in shallow crystallizing dishes. Filter paper cones covered the dishes to prevent any dust from the air entering the solution. This process gave a quantity of large transparent crystals $CdSO_4.8/3H_2O$. These were dissolved in pure water so that a saturated solution resulted after several hours of rapid stirring with an excess of crystals at room temperature.

## FILLING THE CELL.

As has been stated in the description of the cell blank, Fig. 1, the central chamber is made the cathode while the outer concentric tube is made the anode. The cadmium amalgam is introduced through the circular opening near the top of the cathode tube by means of a smaller glass tube which is curved so that all the amalgam enters the chamber. A depth of 1.5 cm. is sufficient to amply cover the tungsten lead. The spherical part of the leg of the cathode chamber is filled slightly more than half full with mercury. The paste, as described above, is carefully pipetted into the cathode chamber so that it covers the mercury to a depth of about 2 cm. Two methods are used to hold the mercury and paste in position. The first method which has been used heretofore consists in thrusting a silk-covered hollow cork ring down upon the surface of the paste. The opening through the hollow cork is about 0.5 cm. in diameter. To insure the plug from chemically affecting the E. M. F. of the cell, it is first thoroughly boiled in water, dried free from moisture and allowed to soak several days in saturated cadmium sulphate solution. Acid treated silk which has been thoroughly washed and dried exists

in a stable condition in contact with the cell ingredients. The alternate method of holding the paste and mercury in position consists in using a clean coil (two turns) of No. 28 (0.32 mm.) tungsten wire, which is slightly larger than the cathode tube to replace the cork used above. A slight annular concave depression made in the cathode tube when constructing the blank serves to hold the silk and coil in the proper position.

The cell was next filled to the upper seal with the saturated cadmium sulphate solution in which a few crystals of clear




CURVE 1. Saturated H-Type Standard Cells—Hard Glass Tungsten Leads.

$CdSO_4.8/3H_2O$ have been added. By placing the cell in cold tap-water to a depth of 6.5 cm. a smooth seal may be made by judiciously softening the hard glass with the temperature of the oxygen-natural gas flame. Before sealing the cell off permanently, a small capillary opening is left at the top of the seal and is closed after the glass above the tap-water has reached the room temperature. This frees the interior of the cell from any reduced pressure effect. No basic cadmium sulphate is thrown out of solution when these precautions are taken in sealing off the cell.

## ELECTRICAL TESTS.

After completion, these cells were tested periodically to determine the constancy of the E. M. F. A Wolf potentiometer was used for this purpose. Six portable unsaturated cells of the Weston type served as standards. Four of these cells were sent to the Bureau of Standards for check at various times and a weighted mean of the certificate values of these six cells was used in checking the new cells. The cells were not placed in an oil bath but before checking they were kept in the standardization room for several hours and the air temperature close to the cells noted. The temperatures were accurate to at least one-half degree. Curve No. 1 shows a series of tests covering a period of 2½ years for an early set of saturated H-type cells made up of hard glass and tungsten leads. These cells show a smaller variation of E. M. F. than do the individual portable cells used for standardizing them. Table I gives a record of the E. M. F. of a set of unsaturated cells of the concentric type. These have not been kept for a long enough period to give useful data on their constancy.

*Table I.*

| Cell | E. M. F. |
|---|---|
| 900 | 1.01822 |
| 901 | 1.01821 |
| 902 | 1.01824 |
| 903 | 1.01813 |
| 904 | 1.01821 |
| 907 | 1.01810 |
| 908 | 1.01815 |
| 909 | 1.01811 |
| 910 | 1.01814 |
| 911 | 1.01824 |

## FACTORS AFFECTING THE STABILITY OF STANDARD CELLS.

Such changes noted at the mercury surface as the hydrolysis and formation of basic salt in the solution give an increase of E. M. F. as the mercury concentrates. Crystalline basic salts stop increase of free acid formation due to hydrolysis, which has a tendency to lower the E. M. F. The E. M. F. is also decreased by evaporation of the liquid, by the change in the crystalline state of the cadmium sulphate and by the inequality of the grain sizes of the mercurous sulphate. It is obvious that the cell blanks must be absolutely clean and the chemicals used must be of excellent

purity. A cell which is very slightly acid makes a satisfactory working standard. A slight variation in the composition of the cadmium amalgam has no harmful effect upon the E. M. F. The tungsten leading into the interior of the cell legs is usually covered with a small quantity of tungstic oxide. This is reduced electrolytically in dilute $H_2SO_4$ at the cathode which leaves a very thin layer of spongy tungsten about the wire. Mercury is deposited upon the tungsten immediately by adding a small quantity of mercurous nitrate to the electrolyte. It is probable that a slight amalgamation of the tungsten is produced in this case. In emptying the electrolyte from the blank and after subsequent washings with distilled water, the tungsten leads remain bright due to the electrolytically deposited mercury. This process of amalgamation of the leads gave a very stable contact with the cell and is free from high resistance effects, which accompany untreated electrodes.

## ADVANTAGES OF THE PORTABLE CELL.

The portable cell as described has at least five distinct advantages:

(1) Compactness.

(2) The cathode is centrally located, holding the mercury in a central chamber.

(3) Stability of construction.

(4) Legs are kept at the same temperature, thus reducing E. M. F. variations.

(5) Constant electromotive force is obtained over long periods of time.

The authors wish to express their thanks to James G. Ford and Paul O. Askin for assistance in setting up and testing many of the cells.

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*A paper presented at the Thirty-eighth General Meeting of the American Electrochemical Society, in Cleveland, October 1, 1920, President Landis in the Chair.*

# INDUSTRIAL APPLICATIONS OF ELECTROLYTIC CONDUCTIVITY MEASUREMENTS.[1]

By Earl A. Keeler.[2]

ABSTRACT.

Use of accurate conductivity measurements on electrolytes, to determine therefrom variations in their composition, such as using a salt solution of known conductivity as a solvent for sugar, and determining the amount of sugar by its effect in increasing the resistivity of the solution. Such measurements may be useful methods of control in chemical manufacturing operations, several of which are suggested.—[J. W. R.]

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Recent advances in the methods of making conductivity measurements have made possible the practical application of this type of measurement to the recording and control of industrial processes. In many instances uniformity of product, along with considerable saving of time and money, may be effected by the use of such measurements. The satisfactory results secured by utilizing the new method, coupled with the many possibilities for further application to the industries, make the subject well worth considering. Accordingly, this paper is presented with the expectation that it may be of interest and assistance to those who are considering the practical application of conductivity measurements.

The usual method of making conductivity measurements requires the use of a Wheatstone bridge with a high frequency source of alternating current and a telephone receiver as a detector. For absolute measurements of conductivity this method

[1] Manuscript received November 24, 1919.

[2] Research Department, Leeds and Northrup Co., Philadelphia.

is unquestionably the best. It has the advantage of being practically free from polarization errors. Its disadvantages lie in the necessity for quiet surroundings and an operator possessing a fair degree of skill. For industrial work the polarization errors are not important, since it is usually sufficient to base the measurements upon comparisons rather than upon absolute values of conductivity. It is only important that the polarization errors remain practically constant under similar conditions. This permits the use of low-frequency current, which can be conveniently obtained from commercial alternating current supply circuits. Thus the necessity for a somewhat troublesome high-frequency generator is eliminated. By substituting for the telephone receiver a simple alternating current galvanometer, which differs only slightly from an ordinary portable D. C. galvanometer, the necessity for a skilled operator is also eliminated. It is much easier for an unskilled operator to balance a bridge by means of a galvanometer and low-frequency current than by use of the telephone receiver and high-frequency current.

The greatest advantage, however, of the low-frequency method is to be found in the ease and convenience with which it lends itself to the construction and practical operation of automatic recording and control apparatus. If recording and control are not necessary, a convenient and portable hand-operated bridge may be used. The alternating current for the apparatus is then secured by connecting with a flexible cord to commercial supply circuits. The conductivity cell is usually mounted in a metal case to avoid breakage, and, when not in use, is conveniently placed in a compartment provided for that purpose.

As a matter of fact, the construction of practical measuring apparatus has ceased to be a source of great difficulty. The greatest difficulties in the application of the method usually arise from temperature variation of the liquid and the design of the conductivity cell.

Temperature variation can be satisfactorily taken care of by automatic compensation in several ways. The cell design, however, requires careful attention. Breakage must be reduced to a minimum without sacrificing the electrical insulation properties. Sufficient circulation of the electrolyte through the cell must be secured in order that the variations of conductivity may be fol-

lowed accurately. The design should also be such as to facilitate cleaning and inspection. A rugged and "fool-proof" cell will eliminate most of the difficulties that arise in conductivity measurements under industrial conditions.

Conductivity measurements cannot, as a general rule, be applied as a means for analysis of solutions. It is usually impossible to distinguish between the various salts which may be in solution together, although if one salt is materially higher in concentration, mobility of its ions, or degree of dissociation, its concentration may be determined in the presence of other salts. It is also important that the concentrations of the other salts present remain

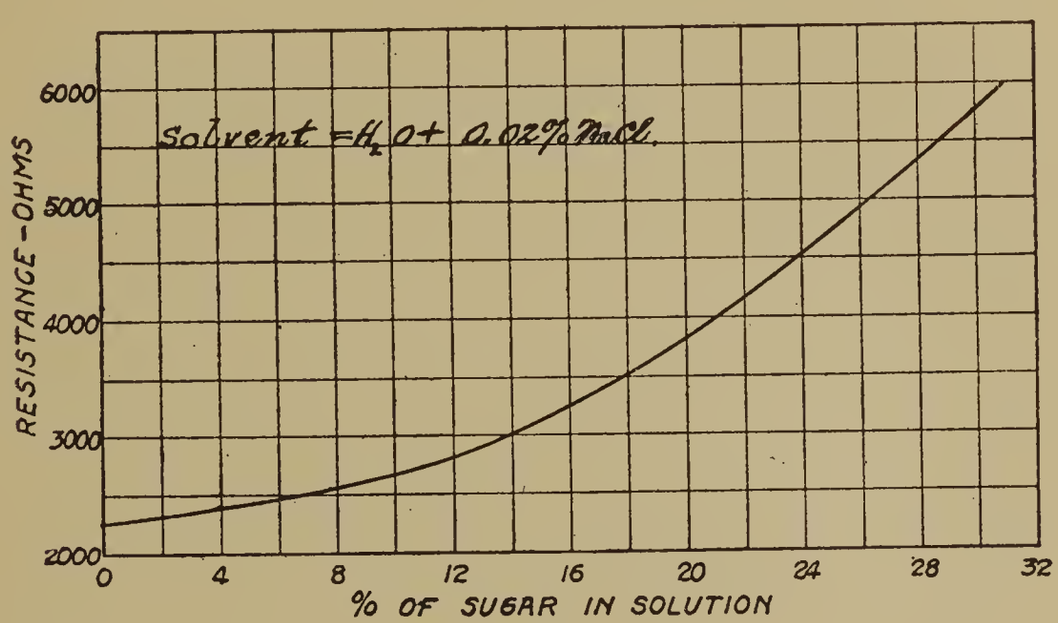


Fig. 1.

constant or nearly so. The portion of the conductivity due to the salt to be determined will then largely preponderate over that due to the other salts present. In any event, variations of the concentrations to be determined should practically be the sole cause of all the changes of conductivity that take place. The accuracy of the measurement necessarily depends upon the degree to which this condition can be secured.

It is not always well to base a decision on purely theoretical grounds, unless one is very careful to take into account the other conditions of the problem. For example, consider the application of conductivity methods to the determination of concentrations in sugar solutions. It is well known that the degree of ionization in sugar solutions is very small, and consequently the conductivity

method would seem impracticable as a means of measuring the concentrations. This, however, is only true under certain conditions. It is possible to control the conditions so that the changes of sugar concentration produce material changes in resistance. This may be done by utilizing, as the solvent, water that already contains a small amount of soluble salts. The solvent then has a definite conductivity, and additions of sugar result in a decrease of conductivity, or increase of resistance, as shown in Fig. 1. (The solvent contained 0.02 percent of sodium chloride.) Fig. 2 shows

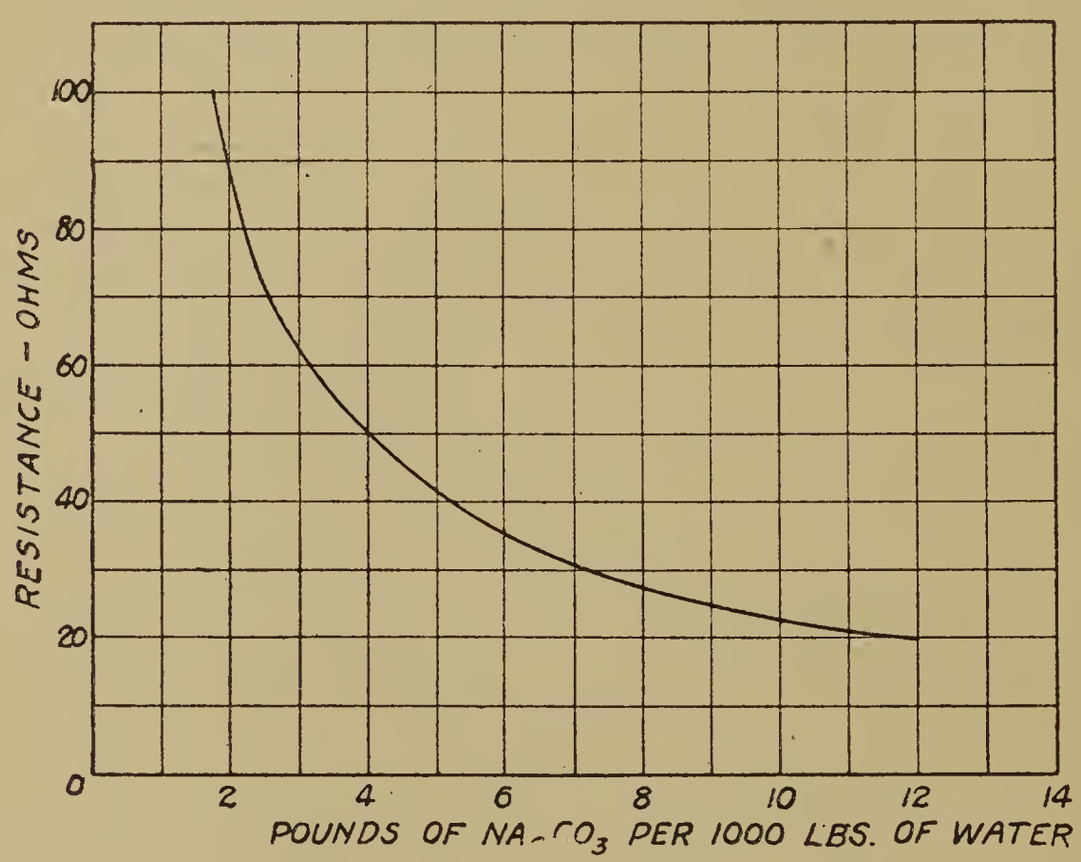


Fig. 2.

the usual type of resistance-concentration curve. The change of resistance is practically entirely due to the interference effect of the dissolved sugar. That is, the un-ionized sugar in solution reduces the average velocity of the ions by obstructing their free passage through the solution between electrodes. It is evident, therefore, that the resistance change is of a nature not usually associated with a concentration-conductivity measurement. A noteworthy application of this type of control is found in the evaporation of sweetened milk. Here the large amount of sugar is effective in reducing the conductivity of the milk, which is largely due to dissolved salts.

Although it is impossible to cover fully the possibilities of the conductivity method, the following list is representative of the wide field of applications. Many of these applications have already been successfully made under industrial conditions. The remainder are so far advanced in the development stage that there is little doubt but that they will prove satisfactory:

(1.) The measurement of boiler water concentrations and the control of the blow-down point.

(2.) Determining the "finish" point in the evaporation of condensed milk.

(3.) The measurement and detection of surface condenser leakage.

(4.) The detection of losses in the tail-races of evaporators.

(5.) The measurement of $CO_2$ in flue gases.

(6.) The measurement and control of acid concentrations.

(7.) The determination of the concentrations of electrolytes in liquid rheostats.

(8.) The measurement of evaporator concentrations.

(9.) Determinations of the salinity of ocean or river waters.

In conclusion, it should be noted that not all chemical processes can be followed by conductivity measurements. For this reason, attention is called to another method, which is particularly well adapted to measurements involving changes in acidity or alkalinity. It is well known that many chemical processes or reactions can be followed and investigated by the measurement of the potential difference or changes in potential difference between two electrodes immersed in the solutions involved. These potential changes result from the changes in magnitude or nature of the ionic concentrations in the solutions undergoing the reaction or chemical changes. The determination of hydrogen ion concentration by means of the potential difference existing between a hydrogen electrode and calomel cell is the most notable example of this type of measurement.

While the technique of the hydrogen ion method has been highly developed during recent years, considerable work must be done to develop and design apparatus that will be satisfactory under the severe requirements of industrial use. Development work

along this line is progressing rapidly, and it is expected that material will soon be available for a paper on the "Industrial Applications of Hydrogen Ion Determinations."

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## DISCUSSION.

W. S. LANDIS[1]: A rather interesting commercial development of the application of electrolytic conductivity measurements was used at the Government nitrate plant at Muscle Shoals. In the pumping of nitric acid over the absorption towers, we used air lifts at the bottom of wells 120 feet deep. There were 110 of these air lifts provided, and handled nitric acid up to 50 percent strength. The acid was carried in the wells through aluminum piping, coupled by flanged joints. The great problem was to determine leakage of these joints in the numerous wells.

The wells were flooded with water and a very small stream introduced at the bottom and permitted to overflow at the top. A pair of very small electrodes connected with a standard voltage lamp connected to the lighting circuit of the plant was inserted in the overflow of each well. The leakage of any joint would permit acid to escape into the overflow water, and by the glowing of the lamp we could detect as small an amount as one-fourth of one percent of nitric acid in this waste water.

E. R. MORTON[2]: In connection with electrolytic cell measurement, I recently looked over a large number of cells that Leeds & Northrup were exhibiting, and in no case were their cells of such design as to be standardized otherwise than by comparison. That is something that ought to be gone into and a type evolved which will give a quantitative measurement without standardizing each individual cell against a standard maintained in the manufacturer's factory.

CARL HERING[3]: I think the paper itself states that it refers only to relative measurements and not to absolute ones.

[1] American Cyanamid Company, New York City.
[2] New York City.
[3] Consulting Electrical Engineer, Philadelphia, Pa.

E. R. MORTON: In connection with that, the design of the cells that Leeds & Northrup manufacture was not such as to permit the cells to be duplicated; it was largely a question of how clever the glass blower was, and how closely he could work to specified dimensions.

MR. KEEL: I do not think Leeds & Northrup keep a standard for standardizing those cells; you can standardize them to an ordinary normal electrode, and anybody can make that.

E. E. STARK[4]: As an interesting side light on that, in the development of an electrolytic heater I found out that by putting ordinary soap in water the conductivity decreased for an increase in temperature, and very rapidly at that.

E. A. KEELER *(Communicated)*: In his discussion of the paper, Mr. E. R. Morton apparently makes the following assumptions:

(1) That the method of standardizing or determining the constant of conductivity cells involves the use of a standard cell maintained in the manufacturer's laboratory.

(2). That duplication of cell characteristics can not be obtained excepting by cleverness of a glass-blower.

Both of these assumptions are incorrect, as may be seen from the following explanation:

(1) A conductivity cell may be checked at the time of purchase and at any other time by a measurement of its constant. The constant is a factor depending upon the dimensions of the cell, and can be conveniently determined by measuring the resistance between cell electrodes when surrounded by a standard solution such as 0.1 *N* KCl. The conductivity at various temperatures of standard solutions can be obtained by reference to Kohlrausch. A simple calculation will then permit determination of the cell constant.

$$\text{Constant} = \text{Conductivity} \times \text{Resistance}.$$

It is evident, therefore, that a conductivity cell can be checked anywhere and by anyone who can make up a known concentration solution of KCl to a reasonable degree of accuracy.

(2) In regard to discussion concerning the duplication of cells, I wish to call Mr. Morton's attention to the fact that the Leeds &

[4] Electrician, Elyria, Ohio.

Northrup Co. manufacture two general types of cells. One type is made of glass for laboratory work, and the other of metal with porcelain insulation for the electrodes. In general, the metal cells are recommended for industrial use, and can easily be adjusted for cell constant by varying the distance between electrodes and using a standard solution in the resistance determination. As a matter of fact, it is a simple matter to produce any number of cells with cell constants agreeing within a fraction of 1 percent.

*A paper presented at the Thirty-eighth General Meeting of the American Electrochemical Society, in Cleveland, October 2, 1920, President Landis in the Chair.*

# LEAD PLATING FROM SODIUM HYDROXIDE LEAD BATHS BY THE USE OF ADDITION AGENTS.[1]

By Frank C. Mathers[2]

## Abstract.

Many addition agents belonging to the class of organic fatty acids or substances such as gums and rosins containing similar acids were found to greatly lessen the formation of loosely adhering crystals on the lead deposits from baths of lead acetate dissolved in excess of sodium hydroxide. Probably gum sandarac and gum galbanum were best, but rosin and oleic acid were quite good. While the deposits were apparently non-crystalline, uniform and dense, corrosion tests in sulphuric acid showed that they offered less satisfactory protection to the metal beneath than lead deposits made in the fluosilicate bath.

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## *Introduction.*

Many attempts have been made to plate lead from alkaline baths prepared by dissolving lead oxide or nitrate or acetate in excess of sodium hydroxide. Such baths have the advantage over the lead fluosilicate bath that they may be quickly prepared from materials that are easily obtained. However, the bath is a failure due to the very crystalline nature of the deposit. The directions for the operation of this bath[3] say that " in order to produce a dense deposit of some thickness, the objects have to be frequently scratch-brushed."

Apparently some attempts have been made to prevent the crystalline structure by the use of addition agents, as is shown by

[1] Manuscript received August 5, 1920.

[2] Associate Professor of Chemistry, Indiana University.

[3] Langbein-Brandt, "Electrodeposition of Metals," p. 472.

the sale of such a bath containing molasses, by a plating-supply company. This bath when tested in our laboratory did not give a satisfactory deposit.

The purpose of this research was to try various addition agents, in the hope that the alkaline lead bath could be made to give as satisfactory deposits as the acid lead baths.

### *Apparatus and Methods of Testing.*

Baths of 200 cc. (0.4 pint) were used. The composition of the baths was 20 percent or grams per 100 cc. (26.7 oz. per gal.) of sodium hydroxide and 7.3 percent (9.7 oz. per gal.) of crystallized lead acetate. This quantity of lead acetate is equivalent to 4 percent (5.3 oz. per gal.) of lead. The temperature was 85° C. (185° F.). The higher the temperature the better the deposits. Two lead anodes were suspended on opposite sides of the beaker by iron wires, and an iron cathode, freshly cleaned in acid, was suspended between the anodes. A current of 1.7 amp. per sq. dec. (15.8 amp. per sq. ft.) was used. The addition of more sodium acetate was found to have no influence upon the deposits, hence no effort was made to prepare baths without this by-product.

The selection of the above concentration was the result of a few preliminary experiments on variation in the concentration of the bath. Baths containing 1 and 2 percent of lead and 5, 10 and 20 percent sodium hydroxide gave brighter deposits the higher the percent of sodium hydroxide. A series of experiments using 20 percent sodium hydroxide and 1, 2 and 4 percent lead gave deposits that were less fluffy and more solid as the concentration of lead increased; then with 4 percent of lead and 15, 20, 30 and 40 percent of sodium hydroxide, the better deposits were obtained from the baths containing 15 or 20 percent sodium hydroxide.

### *Experiments with Addition Agents.*

Most of the addition agents that were tried dissolved fairly well when added directly to the warm alkaline baths. In most cases, 0.5 percent (0.66 oz. per gal.) of the solid addition agent was added; increased quantities of the addition agents were tried but in general these increases produced no material improvement in the deposits.

The following substances produced no improvement in the deposits or at least very slight improvement when compared with the more effective substances: casein, cane molasses, phenol, tannic acid, gum arabic, clove oil, coal tar pitch, lanolin, stearic acid, aniline, nitrobenzene, water extract of pine wood, aloes from Barbadoes, brown sugar, creosote oil, tar oil, starch, gum mastic, eucalyptus, gum gambir, balsam tolu, Burgundy pitch, gum tragacanth, manna, gum guaiac, balsam copaiba, cottonseed oil, turpentine and thin tar oil.

The following inorganic substances showed no value as addition agents: sodium cyanide, sodium silicate, sodium aluminate, sodium thiosulphate ("hypo"), sodium sulphite, arsenious acid, and borax. Relatively large quantities of these substances, as high as 5 percent in some cases, were tried. The strong odor of ammonia from the bath containing sodium cyanide showed that the use of cyanide in these very hot alkaline baths was impossible even if a beneficial action had been observed.

Casein was decomposed by the hot alkaline liquid with evolution of ammonia. The cane molasses had some beneficial influence, as shown by the fact that the crystals were heavier and more firmly attached.

The following substances showed very marked beneficial influence upon the deposits: glue, oleic acid, whale oil, linseed oil, sweet almond oil, refined tar oil, sandarac, gum benzoin, Canada turpentine, gum rosin, euphorbia, gum galbanum, shellac, gum thus, gum ammoniac and asafetida. Of the above addition agents the sandarac, galbanum, euphorbia and rosin were perhaps the best. Probably others of the gums and rosins would have given good results if they had been tried. None of these addition agents dissolved completely in the alkaline baths. Even the soaps from the rosin and the oleic acid only partly dissolved. The limited solubility of some of the gums and oils may have been the only factor that caused them to give unsatisfactory results. This was undoubtedly true in the case of stearic acid. Some difficulty was caused by the insoluble matter, coming both from the impurities in the anodes and from the addition agent, making the cathodes rough. This trouble would not have been so great in cold baths but the high temperature kept the sludge thoroughly mixed throughout the baths.

The addition agents dissolved better in baths using potassium hydroxide in place of the sodium hydroxide, but the deposits were not sufficiently better to warrant the change.

Sandarac was probably the best addition agent, but good deposits can be obtained with rosin. About 0.3 to 0.5 percent of sandarac and 0.5 to 1 percent of rosin should be used. It is best to dissolve the addition agents in dilute sodium hydroxide before putting them into the baths. A precipitate always forms, but if less addition agent is used in order to avoid this precipitate less good deposits are obtained.

### *Tests of Lead-plated Iron in Sulphuric Acid.*

In all cases, iron objects plated in these baths were more readily attacked by 5 percent sulphuric acid than were similar objects plated in an acid lead bath. There seemed to be pin holes in the deposits through which the acid reached the iron.

### *Current Efficiencies at the Cathode and the Anode.*

A bath containing 4 percent lead, 20 percent sodium hydroxide and 0.5 percent of rosin gave a cathode efficiency of deposition of 99.6 percent and an anode efficiency of solution of 73.9 percent, with a current density of 1.75 amp. per sq. dec. (15.8 amp. per. sq. ft.) at both electrodes. At room temperature, the anode efficiency was only 56 percent. With the anode surface increased so that the current density was 0.35 amp. per sq. dec., the anode efficiencies were 99.3 and 100.9 percent; at 0.5 amp., 99.6 and 99.3; at 0.7 amp., 99.4 and 101.4; at 1 amp., 99.4 and 99. These data show that the current density at the anode should not be greater than 1 amp. per sq. dec. (9.3 amp. per sq. ft.) and that the high temperature is necessary for proper anode corrosion as well as for improvement of the cathode deposit.

## SUMMARY.

1. Gum sandarac, gum galbanum, rosin and oleic acid were satisfactory addition agents for restraining the crystalline structure of the cathode deposits in alkaline lead baths. Other gums, rosins and fatty acids showed marked beneficial influences.

2. A satisfactory concentration of bath is: 7.3 percent (9.7 oz. per gal.) of crystallized lead acetate, 20 percent (27 oz. per

gal.) of sodium hydroxide and 0.3 to 1 percent (0.4 to 1.32 oz. per gal.) of addition agent such as gum sandarac, rosin, etc.

3. As high a temperature as possible should be used. Better deposits are obtained at 90° C. (194° F.) than at 80° C. (176° F.).

4. The current density at the anode should not be greater than 1 amp. per sq. dec. (9.3 amp. per sq. ft.). A cathode current density of 1.5 to 2 amp. per sq. dec. (14.1 to 18.8 amp. per sq. ft.) can be used, but the lower the current the better the deposit. At these current densities, the corrosion and deposition are approximately 100 percent.

5. The deposits are not as perfect as those from the acid lead baths, hence they are less satisfactory as protection against sulphuric acid.

6. Sludge in the baths at these high temperatures tends to cause roughness of the deposits.

7. This research is of importance in relation to the general subject of addition agents, in that it shows a class of addition agents that successfully overcome the crystalline structure in deposits from alkaline baths.

*Indiana University,*
*Bloomington.*

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## DISCUSSION.

F. C. Mathers: This paper gives the results of experiments with addition agents in alkaline lead plating baths. These samples show some typical deposits. The first one is from a bath containing no addition agent. The loose crystalline deposit is characteristic of such baths. These other samples are from baths containing gum sandarac, oleic acid and rosin respectively. The great smoothness of these deposits shows that there is no trouble with crystalline structure. However the deposits are not as impervious as deposits from the acid lead baths and, therefore, are

not so good as protective coatings. I do not feel that they are of commercial value. This research is of value only in that it shows a new class of addition agents for alkaline baths where successful addition agents were not known. Some years ago an alkaline lead bath containing an addition agent, which was probably molasses, was sold on the market by a plating supply house. I never obtained any satisfactory deposits from it.

COLIN G. FINK[1]: A good deal has been done on lead plating and lead refining. I had occasion a little while ago to review the work of Anson G. Betts, a member of our Society. He tried out very carefully a number of lead electrolytes, and finally came to the conclusion that the lead fluo-silicate solution was the best electrolyte to use. It occurs to me, that here is a good chance for a university laboratory, Indiana University, for example, to go over this work of Mr. Betts and check up the adaptability of some of the simpler electrolytes. It is well known that lead chloride and lead sulphite are insoluble, but there are other soluble lead salts a good deal cheaper and simpler than the fluo-silicate. There is a very good opportunity for the university to carry out small scale experiments along this line, assuming that the fluo-silicate bath is not the final answer.

F. C. MATHERS: I would like to ask what soluble salts the speaker has in mind?

COLIN G. FINK: I'd rather have you start out and try yours. I will be better prepared to say in another year.

W. S. LANDIS[2]: There is a very important phase of this lead depositing and lead plating that is interesting others than strictly lead platers. The cost of lead lining for tanks is high, and lead burners are becoming scarcer every day. While platers are working on these thin coatings, a hundred-thousandth of an inch thick, somebody should work on obtaining an eight or ten-pound (to the square foot) lead coating, so that we could get away from the lead burner.

WM. BLUM[3]: In that connection I would call attention to a paper presented last year on the use of fluoborate lead plating baths, in

[1] Head of Laboratories, Chile Exploration Co., New York City.
[2] American Cyanamid Co., New York City.
[3] Chemist, Bureau of Standards, Washington, D. C.

which there were no original developments so far as composition was concerned, but in which it was shown that it is perfectly possible to deposit lead up to 0.05 inch or more in thickness, on the inside of chemical apparatus. Such work had actually been started for the Edgewood Arsenal at the time of the armistice; and I think that anybody interested would be justified in looking into the commercial applications of lead plating. Recently we were asked whether it was possible to plate the inside of a tank car with lead. I will not say that this is practicable, but the fact remains that there are great possibilities in lead plating.

C. P. MADSEN[4]: I would like to ask Dr. Mathers whether his work with colloids and addition agents is based on any scientific theory or whether his work is simply empirical experimentation.

F. C. MATHERS: Part of these discoveries of addition agents are accidental. After one locates a family of materials, then, of course, it is easy enough to pick out the particular one that is best, but in the beginning it is largely a matter of "cut and try."

A. L. FERGUSON[5]: In this connection, that is the point that interests me very much. There has been much work done recently on the subject of addition agents, and it is coming to be a very important factor in plating. I should like to ask Dr. Mathers or anyone else if they have any explanation or can account in any way for the action of these addition agents? How is it they do smooth out the deposit and make it better? How do they act? And then, in line with the remarks of the previous speaker, that would help us, I think, in choosing what addition agents to use; that is, it would not be a case of making a haphazard guess, but one would have some scientific basis for the selection. I know the colloidal theory has been offered by some, but it has not been extended to the point where it is satisfactory, as far as I know.

F. C. MATHERS: I do not know why these act as they do. Dr. Bancroft insists it is colloidal adsorption whereby the size of the crystals is reduced. That is as good an explanation as I know of.

COLIN G. FINK: There are a number of theories that have been advanced in regard to these addition agents and their effects. There are some metals, such as tin in a tin chloride bath, which

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[5] Instructor in Physical Chemistry, Univ. of Michigan, Ann Arbor, Mich.

without addition agents readily form very long crystals or needles at the cathode. Now, if you add to that tin chloride bath a colloidal material which will remain in suspension in presence of the electrolyte, what happens is that as soon as a small crystal of tin begins to form on the cathode, there is apparently an attraction for the colloidal particles which attach themselves to the points of the crystals. What we observe under the microscope is that the little tin crystal does not grow in the accustomed straight line, but a new small crystal forms on its side: the original path is blocked, apparently. Every new tin crystal in turn attracts to its point a small colloidal particle, and on the side of that crystal a new crystal grows, so that instead of having one long crystal, as happens without addition agents, we get a fern-like structure or fine crystal conglomerate.

There is another type of addition agent that tends to form complex salts with the metal ions; it is to some extent similar to the complex silver sodium cyanide. In the case of silver sodium cyanide you throw out sodium as your primary product, which in turn reacts upon the complex silver cyanide ion and throws out silver as a secondary product. Primarily deposited silver will always, or in almost all cases, be coarse crystalline, whereas secondary silver is very finely crystalline or smooth. These are merely conclusions drawn from practical observations.

In conclusion, I think it would be a good idea for the Secretary to get in touch with Dr. Kern and ask him to line up the costs of the various processes that he proposes for the industries. Dr. Kern is giving us a practical paper on electrolytic tin refining. If Dr. Kern knows of a cheap source of peptone we should like to hear it.

Carl Hering[6]: I was about to offer the same explanation that Dr. Fink has just offered for the prevention of the growth of crystals, but I would like to add something which Dr. Fink did not say. I have studied the action of some of these colloids under the microscope, and I notice that they migrate and deposit just like the electrolytic ions do; most of them migrate toward the cathode, and instead of this being an accidental deposit, they actually are deposited around the crystal by the current; they grow in large

[6] Consulting Electrical Engineer, Philadelphia, Pa.

groups around the end of every crystal, because the current density is greater there; this stops a crystal from growing, even when it is only microscopically small. Some particles migrate with the current and some against it, and therefore it seems to me to be important, in the selection of such colloids, to choose only those which travel with the current, that is, which will deposit on the cathode instead of on the anode. I have examined dozens of them, and most of them travel with the current; very few travel against it.

I might take this opportunity to refer to the paper I read about a dozen or more years ago describing this research. With some difficulty I found, in the same solution, one of those colloids which travels in one direction and one which travels in the other, and I arranged the microscopic slide so as to get two of these particles into such a position that they would travel toward each other, as I was interested to see what would happen when they met, because evidently there is a positive electrification on one and a negative on the other. I thought that they would have an attraction for each other and would combine, but when they nearly met they turned out of each other's way like two wagons in a narrow street. Apparently, the adsorbed coating around them more than resisted the electric attraction, as is apparently also the reason why in the Brownian movements the small oscillating particles never touch each other.

Wm. Blum: Just one other point. We were just told that in some way these colloidal particles are attracted and later actually deposited. We can go a little further and state an obvious reason why there should be an accumulation of these colloidal particles, viz.: that upon any point or projection there is a higher current density than on any other part of the surface, and therefore there must be a greater proportion of these colloidal particles carried to that point than to other portions of the surface. We have, therefore, the theory of the movable diaphragm, that from time to time this accumulation of colloidal particles becomes sufficient to produce an effective diaphragm, or an increased resistance at least, and the current goes to some other point until the current density on that point reaches a magnitude which will cause a large number

of colloidal particles to deposit. That theory is, I think, what Mr. Fink and Mr. Hering both had in mind.

The question has often been raised as to whether all the organic addition agents serve as colloids. As pointed out a year ago, it is probable, although by no means actually proven, that the addition substances are either colloids or give rise in solution to colloids. The substances which are, by nature, reducing agents rather than colloids, such as hydroxylamine, etc., probably produce colloids of some kind. In the paper a year ago reference was made to the work upon the silver coulometer, which proved that even very minute amounts of certain substances produce an increase in the weight of the cathode deposits, which is indisputable evidence that under such conditions there is material carried to the cathode along with the silver. If we consider the question of the inorganic addition agents other than those which, as Dr. Fink says, affect the ion concentration of the solution, we have those such as stannic, ferric or aluminum sulphates, which almost certainly can produce colloidal particles. If the objection is raised that in an acid copper solution there is little chance for hydrolysis of aluminum sulphate, we must always remember that in the film adjacent to the cathode there is a tendency for the solution to become neutral or alkaline, and therefore an opportunity for the separation of the colloidal particles.

Some work we have done during the last year has indicated that the prime cause of roughness is the presence of foreign solid particles in the solution, and if you can keep solutions free from suspended matter such as the particles derived from the anodes, there is very little difference between the behavior of certain solutions, whether they contain addition agents or not. The effect of addition agents is, therefore, in a large degree, to overcome the deleterious effect of suspended metallic or other particles of matter in the solution.

Colin G. Fink: Dr. Blum brings out a very interesting point, the clarifying action of addition agents in a number of baths. We must add enough addition agents not only to improve upon the cathode deposit, but to take care of material loosened from the anode which would, in the absence of that addition agent, interfere with a good deposit at the cathode. I would like to put a

question to our addition agent experts; I would like to ask them whether they ever tried to test these addition agents in a cell with a semi-permeable partition, or a porous partition, first putting the addition agent in the cathode compartment to see how it affects the deposit; then, in the next experiment, putting the addition agent in the anode compartment to see how it affects the results? I think there are very interesting surprises in store for those who undertake these experiments.

MR. MORTIMER: I should like to ask these gentlemen if any of them have made an observation of the effect of the addition agent on the electrical resistance of the electrolyte?

WM. BLUM: I can only speak qualitatively on that subject. Statements are frequently made in the literature that the addition of such substances as glue in small amounts changes the electrical resistance of a solution. We have not made any precision measurements, but simply from conductivities measured with a rough outfit for getting what might be called semi-quantitative results, I think that the effects on the conductivity produced by most addition agents are not measurable, certainly they are not of the magnitude of 1 percent. I believe that the use of an addition agent may change materially the voltage required to produce a certain current density, *i. e.*, tend to increase that voltage so that you get effectively an increase in the resistance; but it is not an increase in the resistance of the solution but of the cell.

C. B. HOGABOOM[7]: In the electro-deposition of silver from solutions to which carbon bisulphide has been added, it is necessary, if you are running a normal bath, say of 3 ounces of silver and about 3 to 3½ ounces of free cyanide per gallon, to increase that cyanide content to at least 5 ounces and increase the pressure to at least 1½ volts.

A. L. FERGUSON: We may be able to furnish more information along that line next year. We have at the present time under investigation a subject of that nature, the addition in various quantities, of various substances, such as acids and some other electrolytes to a colloidal solution, and measuring their influences on its conductivity. It is known that gelatine, for instance, has a conductivity of its own, and this was measured by us in connection

[7] Research Electroplater, Scovill Mfg. Co., Waterbury, Conn.

with some other work; it was also shown that gelatine added to some electrolytes has a decided effect on their conductivity. In connection with this matter of addition agents, some five or six years ago a paper appeared by Mr. A. Mutscheller, I believe, on the subject of the influence of gelatine on the deposition of silver in silver nitrate solutions. It is well known that you cannot deposit silver in an adherent form from silver nitrate, although silver nitrate is the solution used in the standard silver coulometer. His work was done more from the plater's point of view than as an investigation of the coulometer. He added various quantities of gelatine to silver nitrate, and the deposit gradually smoothed out; finally he was able to get a perfectly smooth, adherent deposit that would stand washing. This deposit would not be of much value from the plater's point of view, but it is from a scientific point of view, as it can be washed and weighed without any difficulty. He explained the behavior of the gelatine in this way; he said that gelatine had an influence on the transport number. That was really the subject of the paper. We know that part of the current is produced by movement of anions to anode and the remainder by movement of cations to cathode. By the addition of the gelatine, the silver was caused to carry all the current. In fact, he went so far as to state, and gave data to the effect, that the anion also could even be caused to move toward the cathode, that is, both the silver and nitrate ions may move toward the cathode, and he got a deposit of dark colored silver which he claimed contained some carbon which resulted from the deposition of the gelatine along with the nitrate ion. It did not seem logical to me at all, that such a thing could happen, and last year we undertook to repeat this work, (this is advance information, to be published shortly), and we found results contradictory to his, that is we could add any quantity of gelatine, and the transport number remained absolutely the same. This indicates that the addition of the gelatine to the silver nitrate has no effect whatever on the transport number of silver nitrate, and I believe we have proven that point. His explanation was that the gelatine is positively charged, that it is a positive colloid and the nitrate ion having a negative charge was adsorbed by the gelatine, the resulting complexes when rather large quantities of gelatine were used were

themselves positively charged, and moved naturally toward the cathode, so that when he had added sufficient gelatine to combine with or adsorb all the nitrate ions, all the ions were positively charged and moved toward the cathode.

F. C. MATHERS *(Communicated)*: I do not think that any of the theories advanced in this discussion to account for the action of addition agents agree sufficiently well with the mass of the facts. Many addition agents are specific for certain metals and sometimes even for certain salts only of these metals. Very briefly, I think that there must be formed complex cations containing the addition agent and also the acid radical. No such general theory as "migration to the cathode" explains why an addition agent is not effective with all the salts of all the metals. I do not wish to go into a long discussion of this theory at this time.

*A paper presented at the Thirty-eighth General Meeting of the American Electrochemical Society, in Cleveland, October 2, 1920, President Landis in the Chair.*

# TIN PLATING FROM ALKALINE TIN BATHS BY THE USE OF ADDITION AGENTS.[1]

By Frank C. Mathers[2] and William H. Bell.[3]

## Abstract.

Smooth adherent tin deposits were obtained from alkaline tin baths, using balsam copaiba or rosin as addition agents. Without the addition agents the deposits were non-adherent and crystalline or spongy. The serious objection to these baths is that they gradually deteriorate until finally good deposits cannot be obtained.

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## INTRODUCTION.

Alkaline tin baths prepared by dissolving stannous chloride in sodium hydroxide do not give smooth, firm deposits on the cathode. The deposits are non-adherent and crystalline or spongy, and are unsuited as protective coatings. The object of this research was to study the effects of the various addition agents, in the hope of finding one that would produce smooth, non-crystalline deposits. It must be remembered that Beneker[4] used relatively large quantities of sodium thiosulphate ("hypo") in the sodium stannite bath. Tests here have shown that this bath gives good deposits, but that the bath gradually deteriorates, and, after a time, produces an entirely unsatisfactory deposit.

## TESTS OF ADDITION AGENTS.

The preliminary work consisted in the testing of the various addition agents. Each test bath had a volume of 500 c.c. (1.1

[1] Manuscript received August 5, 1920.
[2] Associate Professor of Chemistry, University of Indiana.
[3] Sec'y-Treas., National Stain and Reagent Co., Norwood, Ohio.
[4] U. S. Patent 921,943.

pint). The composition was 6 percent[5] (8 oz. per gal.) of crystallized stannous chloride and 6 percent (8 oz. per gal.) of sodium hydroxide. Two cast tin anodes, 3.7 cm. sq. (1.5 in.), were suspended with iron wires on oppposite sides of a beaker. An iron cathode, freshly cleaned in acid, was suspended between the anodes. The current density was 0.5 amp. per sq. dec. (4.7 amp. per sq. ft.); the temperature 50° C. (122° F.). In each case 0.1 percent (0.13 oz. per gal.) of the addition agent was added. The increase in the addition agent above this amount was not found to appreciably improve the deposit. The solid addition agents were added directly to the warm baths in most cases. If the addition agent did not dissolve well, alcoholic solutions were used.

The results with the addition agents were as follows:

*Sandarac:* Smoother and less crystalline than with no addition agent.

*Rosin:* Smooth and adherent, but a little crystalline on the edges.

*Glue:* Excellent deposit. However, the glue was rapidly changed by the hot alkali, and the products formed caused poor deposits. Further additions of the glue were then of no value. Glue would have been a satisfactory addition agent if it did not decompose.

*Oleic acid:* Smooth, adherent deposit, but with a tendency to become crystalline on the edges. Previous sulphonation of the oleic acid somewhat improved the deposit.

*Gum tragacanth:* Smooth, slightly crystalline.

*Manna:* Smooth, slightly crystalline.

*Aloin:* Non-adherent and crystalline.

*Sodium sulphide,* or *sulphur,* or *sodium sulphide and rosin:* Crystalline and non-adherent.

*Balsam copaiba:* Smooth, adherent deposit.

The balsam copaiba was the most satisfactory of any, and rosin probably ranked next. These substances produced deposits that were sufficiently adherent, smooth and non-crystalline for commercial use.

[5] Percent as used in this paper means grams per 100 c.c. of bath.

DETERIORATION OF THE BATH.

The balsam copaiba or rosin gave good deposits in new baths, but after a time spongy deposits were obtained.[6] The use of new portions of addition agent did not overcome the trouble. The addition of more sodium stannite corrected the trouble, thus showing that the concentration of stannous salt had decreased. This could have been caused either by the oxidation of the stannous salt or by low anode corrosion. Tests with a coulometer in series with the baths showed that only 76 to 78 percent of the theoretical quantity of tin dissolved from anodes made of ordinary commercial tin. A black, hard film formed on the anodes. The addition of 1 percent sodium cyanide to the bath lowered the corrosion of the anode to 55 percent. The addition of sodium chloride or sodium sulphate had no effect on the corrosion. This anode trouble was corrected by using amalgamated anodes made from electrolytically refined tin; three different baths showed anode corrosion efficiencies of 101.5, 102 and 101.6 percent respectively. However, the lives of the baths were not materially greater with the pure than with the impure anodes. This showed that the oxidation of the stannous salt must be the main source of the trouble. Analytical determinations of the stannous salt in some baths which were being run for 8 to 10 hours in each 24 hours showed the following percentages of tin:

| Time | Anodes Amalgamated, Refined Tin | Anodes Commercial Tin |
|---|---|---|
| At start | 2.59 | 2.57 |
| 25 hours | 2.07 | 1.82 |
| 90 hours | 0.11 | 0.11 |

In one experiment the addition of 1 percent sodium cyanide somewhat slowed down this oxidation of the stannous salt, which had dropped to 0.23 only after 216 hours. A bath without addition agent or electrolysis or heating showed a similar decrease in stannous salt, only the rate was slower.

Glycerine, milk sugar, cane sugar and alcohol were added to separate baths with the idea that they might prevent the oxidation in the way that they protect sulphites from oxidation. However,

[6] The rest of the experiments were run at 75° to 90° C. (167° to 194° F.), because the higher temperatures gave deposits that were much superior to those obtained at 50° C.

they showed no beneficial action, and, moreover, hindered the action of the addition agent. Electrolysis of the baths with small iron cathodes did not aid in regeneration. Boiling the baths with sulphites did not regenerate them. No variation in concentration or composition prevented the deterioration.

## SUMMARY.

1. Balsam copaiba, rosin and, to a lesser extent, other similar organic fatty acids or acid-containing substances were found to be successful addition agents in producing smooth, adherent deposits of tin from sodium stannite baths.

2. The composition of the baths was 6 percent[5] (8 oz. per gal.) of crystallized stannous chloride, 6 percent (8 oz. per gal.) sodium hydroxide and 0.1 percent (0.13 oz. per gal.) of the addition agent. A current of 0.5 amp. per sq. dec. (4.7 amp. per sq. ft.) and a temperature of 75° to 90° C.( 167° to 194° F.) were used.

3. These experiments were unsuccessful in that the sodium stannite baths gradually oxidized or deteriorated to such an extent that continued satisfactory deposits could not be obtained. No method of regenerating the baths was found.

4. The use of sodium cyanide in these baths, as is frequently recommended, was of no advantage.

5. This bath is not recommended for tin plating, on account of this deterioration. It would be unreasonable to bother with this sodium stannite bath when one can use the stannous sulphate bath, which gives splendid deposits without any important difficulties.

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*Bloomington.*

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## DISCUSSION.

F. C. MATHERS: These samples of tin plating show the effects of addition agents in baths of sodium hydroxide and stannous chloride. With no addition agent, this cathode deposit is very crystalline and worthless as a protective coating. Many of the

loose crystals have already been broken off by handling. These other smooth non-crystalline deposits were made from baths containing balsam copaiba and rosin, respectively, as addition agents. No indication of crystalline structure can be observed even on the edges and corners. In general, the resins and fatty acids are beneficial addition agents. The great difficulty with these baths is their deterioration during operation or on standing. A precipitate forms and the amount of stannous tin decreases until good deposits can no longer be obtained. This last cathode shows a deposit from the same bath after standing for three weeks; a bath from which one of the best deposits was obtained. This deposit is loose and entirely bad. We found no method of preventing this deterioration. We do not recommend this bath for tin plating because of this deterioration.

G. B. Hogaboom[1]: During the war a tin solution made from tin chloride to which glucose in the form of corn sugar was added was used for plating booster caps. The anode corrosion was not efficient enough, and it was necessary to add a saturated solution of tin chloride. The deposit was heavy enough to resist the action of gases used in gas warfare.

Colin G. Fink[2]: I would like to ask Mr. Hogaboom how thick are the deposits he refers to? When talking about quality of tin deposits I think it is always well to mention the thickness of these deposits.

G. B. Hogaboom: The thickness of deposit required in electroplating has never been definitely settled. A deposit of nickel 0.0004 inch (0.01 mm.) thick is considered a good plate on brass work. The thickness of the deposit is determined by the wear to which the article is to be subjected and the character of the object deposited.

J. W. Richards[3]: I measured, by calculation and weight, the thickness of a copper plating, and it was one seventy-thousandth of an inch (0.00036 mm.) thick; it was a fine copper deposit.

F. C. Mathers: I am sorry that the practical plater finds it necessary to produce so much cheap stuff which does not give

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[2] Head of Laboratories, Chile Exploration Co., New York City.
[3] Professor of Metallurgy, Lehigh University, Bethlehem, Pa.

much real service. I think this statement applies to much of the brass plating. Some of it is called tenement house hardware. A sample of this work in our museum case has rusted and changed until it can now only be recognized as brass plating by its identification card. This failure in most cases is due to the deposits being too thin. It is much easier to produce thin deposits than thick ones. Good-looking thin deposits can be obtained from many baths, but good thick deposits can only be gotten from exceptionally good baths. I do not consider a bath as being satisfactory unless good thick deposits can be obtained from it. Some baths that are used commercially do not come up to this standard.

As regards the continuous operation of the tin bath mentioned by Mr. Hogaboom, I do not consider that this was continuous operation of a bath. The regular addition of tin salts to this bath was equivalent to the preparation of a series of new baths. My idea of a good bath is one that maintains itself for a long time with only unimportant changes.

I want to express my appreciation of the very great help which Mr. Hogaboom has always given to me in all of my researches and problems. He has always cheerfully and very fully answered all of my questions. On the other hand, it would help much if we could influence him to write some of his great fund of information in papers so that it might be available at the beginning of the researches. Too much of his information is *post mortem* so far as the research itself is concerned.

G. B. Hogaboom: I would like to defend the plater. He does not control the thickness of deposited metal. When an article is made and required to be electro-plated with any metal, the manufacturer states the thickness of the deposit required. This is determined, to a great measure, by the standard of quality which the manufacturer wishes to set for his particular line, and also upon the price that he can obtain for the article. In other words, the price is generally set by the public as they will not pay the higher price for many of the articles that would be necessary if a very heavy deposit was put on the work. Another important factor is the design. Many articles have a very fine

line design, and should a heavy deposit of any metal be put upon such an article, the beauty of the design would be entirely lost. The work for college laboratories is so small that the manufacturer cannot have their standards as his, but if those purchasing goods for the college laboratories would request a heavy deposit and state to what conditions the article is to be subjected, a deposit heavy enough to withstand such conditions could be had.

As platers we are ready to freely give of our knowledge. I am no prophet, nor even the son of a prophet, and therefore cannot anticipate anything that Professor Mathers might be going to do, and should he try to bring out something that would not withstand the necessary criticisms, unfortunately a *post mortem* would be necessary.

W. S. LANDIS[4]: I think another comment might be made on that; Mr. Hogaboom is out to make money, the usual college is not, and I presume if we compare the operation of most colleges along the same lines as we operate most corporations, we could draw that lack of parallelism to a much greater extent than concerns mere electroplating.

C. P. MADSEN[5]: I wish to comment on the discussion of all of the coating papers presented today. It seems to me that electro-chemists and practical electro-platers are judging these papers from two different points of view.

I can state from experience that these points of view can be reconciled, and that it is profitable to do so. In order to do this we should first realize that coatings may clearly be divided into two classes: decorative and protective. It is true that all decorative coatings would be more useful and desirable if they could also be made protective, but electro-chemists make a mistake, I think, in assuming that because a metal coating is not protective, it is therefore not useful. Many very beautiful decorative coatings on indoor objects are made by platers, and I think it would be profitable for any electro-chemist to study the commercial importance of this art before he makes comments on the desirability of a proposed coating process which is decorative but not protective.

[4] American Cyanamid Co., New York City.
[5] Consulting Engineer, New York City.

On the other hand, however, I also think that electro-platers make a mistake in minimizing the value of electro-chemists in trying to develop means for making all coatings protective as well as decorative. To contend that the development of a stable process for making brass coatings which are only decorative is not worth while, is a mistake; although platers should equally admit that it would be more desirable if such a process could also make a protective coating. I therefore think, in view of the fact that it has been well-known how to make decorative coatings of tin, that Dr. Mathers' efforts to develop a method for making protective ones are well worth while. I furthermore agree with Dr. Mathers that only an electro-deposition process which will deposit up to 1/16th inch (1.6 mm.) thick or more of metal without disturbing the balance of the bath can be depended upon for making truly protective coatings.

*A paper presented at the Thirty-eighth General Meeting of the American Electrochemical Society, in Cleveland, October 2, 1920, President Landis in the Chair.*

# THE ELECTROLYTIC REFINING OF TIN.[1]

By Edward F. Kern.[2]

A number of different electrolytes have been proposed for use in the electrolytic refining of tin. References which were found in the literature and in patent applications are the following:

1. Brand used an electrolyte containing 9 percent by weight of stannous chloride, and 2.5 percent by volume of concentrated hydrochloric acid. This electrolyte did not find commercial application, due to the crystalline cathode deposit. (Schnabel-Louis, Vol. II, 549; Dammer, Chem. Technology, II, 27 and 324.)

2. French patent No. 435,936 was granted Michaud and Delasson for an electrolyte of tin prepared by dissolving stannous chloride in water, adding sulphuric acid until the precipitated oxy-chloride dissolved, and then adding 1 percent magnesium chloride and 1 percent of boric acid. The tin deposits as crystals from this electrolyte. (Jl. Soc. of Chem. Ind. (1912), **31**, 395.)

3. Mention is made that "beautiful, dense cathode deposits of tin can be produced from electrolytes of sodium-stannous chloride, with low current density and at ordinary temperature." (Bull. Soc. d'Enc. L'Ind. Nat., July, 1912, 28.)

4. Quintaine was granted English patent No. 5,496 for use of an electrolyte of tin chloride and an ammoniacal salt (preferably ammonium chloride), for use in refining tin. Low current density is necessary for solid deposits. (Jl. Soc. Chem. Ind. (1900), **19**, 1121, and Mineral Industry, **9**, 649.)

5. Hollard claimed to have prevented the formation of spongy deposit of tin by using an electrolyte containing 12 g. sodium stannate ($Na_2SnO_3$) and 200 g. sodium sulphate per liter, and electrolyzing at temperature of 80° C. with current density of

[1] Manuscript received August 13, 1920.

[2] Assistant Professor of Metallurgy, Columbia University.

2 amp. per sq. ft. (0.22 amp. per sq. dm.). (Bull. Soc. d'Enc. L'Ind. Nat., July, 1912, 28.)

6. Fischer obtained "satisfactory cathode deposits of tin from an electrolyte of ammonium sulphostannate to which was added sodium sulphite, sodium hyposulphite, or potassium cyanide." (Zeit. Anorg. Chem. (1904), **42**, 363.)

7. Neumann received German patent No. 198,289 for process of producing pure, dense cathode deposits of tin by use of an electrolyte of an alkaline sulphostannate, and electrolyzing at about 70° C. The presence of free alkali is claimed to be beneficial.

8. Claus used an electrolyte of sodium sulphostannate, sp. gr. 1.07, for refining tin. The electrolysis was conducted at 90° C. and with current density of 10 amp. per sq. ft. (1.1 amp. per sq. dm.). All the impurities except arsenic and antimony collected as sulphides in the anode residue; they were deposited with the tin. He found that by using an electrolyte of sodium thiosulphate, made acid with hydrochloric acid, the arsenic and antimony would remain as sulphides in the anode residue. (Schnabel-Louis "Handbook of Metallurgy" (1907), Ed. II, 549; Elektrochem. Zeit., **8**, 168.)

9. O. Steiner was granted U. S. patent No. 890,249, German patent No. 193,528, English patent No. 10,230, and French patent No. 374,116, for use of an electrolyte containing 10 percent by weight of an alkaline sulphide to which is added 1 percent by weight of flowers of sulphur. By electrolyzing at 90° C., with current density of 4.5 amp. per sq. ft. (0.5 amp. per sq. dm.), and potential drop below 0.2 volt tin was dissolved, after which he obtained dense, smooth cathode deposits of tin of high purity (+ 99.9 percent). The amount of tin in the electrolyte reached a maximum of about 2.2 percent by weight, and remained constant so long as the sodium sulphide was kept constant. (Elektrochem. Zeit., May, 1908; Jl. Soc. Chem. Ind. (1907), **26**, 768; Electrochem. and Met. Ind. (1907), **5**, 309.)

10. Sperry was granted U. S. patent No. 874,707 for process of refining tin in a diaphragm cell containing an electrolyte consisting of either sulphate, chloride, fluoride, ammonium acetate, ammonium sulphide, or hydrofluoric acid. The impurities which

accumulate in the electrolyte to be removed periodically by withdrawal of a portion of electrolyte and replacing by pure solution. (Jl. Soc. Chem. Ind., **27**, 343; Electrochem. and Met. Ind. (1908), **6**, 76.)

11. Mennicke used an electrolyte of tin fluosilicate from which the tin was deposited as solid mass, similar to lead from fluosilicate electrolytes. The electrolyte contained 10 g. tin and 10 g. free fluosilicic acid per 100 c.c. solution. Current density of 10 amp. per sq. ft. (1.1 amp. per sq. dm.), and average potential drop of 0.4 volt with electrolyte at 20° C. Presence of lead in the anodes finally caused the cathode tin to deposit spongy. Mennicke was granted U. S. patents Nos. 779,091 and 779,092 for process of refining tin in fluosilicate electrolyte. (Electrochem. Zeit. (1915), **12**, 112, 135, 161 and 180; Zeitschrift für Electrochemie, **12**, 112; Electrochem. and Met. Ind., **4**, 26; Mineral Industry, **14**, 555.)

ORGANIC ADDITION AGENTS IN TIN ELECTROLYTES.

12. Hollis was granted U. S. patent No. 916,155 for refining of tin by the use of tin fluosilicate electrolyte to which gelatine or glue has been added for the purpose of increasing the density of the cathode deposit. Mennicke was granted U. S. patents Nos. 779,091 and 779,092 for tin fluosilicate electrolyte without addition agent. See 11. (Electrochem. and Met. Ind. (1909), **7**, 224.)

13. German patent No. 244,567 to Matuschek is for the use of a concentrated electrolyte of ammonium oxalate saturated with tin ammonium chloride ($SnCl_2.2NH_4Cl$) and an addition of black oak tannin (quercitrinic acid). (Metallurgie (1912), **9**; Jl. Soc. Chem. Ind. (1912), **31**, 440; Zeit. für Elektrochemie (1912), **18**, 790.)

14. Steiner found that when he added a colloid (gelatine) to sodium sulphostannate electrolyte for the refining of tin, the cathode deposit was much improved. (See 9.)

15. Kern and Frapwell conducted a series of experiments on the effect of certain organic addition agents in electrolytes of sodium-stannous chloride, stannous fluoride, and sodium-stannous fluoride (Trans. Amer. Electrochem. Soc. (1913), **23**, 228-232); the results were as follows:

*a*. Sodium-stannous chloride electrolytes contained 8 g. of tin and 5 g. of sodium as chlorides per 100 c.c. Electrolysis at ordinary temperature with current density of 10 amp. per sq. ft. (1.1 amp. per sq. dm.) and potential drop of about 0.1 volt when electrodes about 1.25 inch (3.2 cm.) apart. When no addition agent was present, the deposit formed as a mass of bright needle crystals, which soon short-circuited the electrodes. Three addition agents were tried: gum arabic, tannin and gelatine, each added as aqueous solution. The presence of gum arabic, in proportion of 1 g. in 800 to 1,200 c.c. electrolyte, gave best results, causing the cathode deposit to form as small, bright, adherent crystals, which did not short-circuit the electrodes. The addition of tannin and of gelatine caused the formation of a white, colloidal precipitate in the electrolyte, which did not dissolve; the presence of either of these addition agents in the proportion of 1 g. in 500 to 1,500 c.c. of the electrolyte caused the deposit to form as smaller, bright, needle crystals, not very adherent, whereas when not present the deposit formed as large, bright needles. The tannin was slightly better than gelatine.

*b*. Stannous fluoride electrolyte contained 8 g. of tin as stannous fluoride per 100 c.c. Electrolysis at 10 amp. per sq. ft. (1.1 amp. per sq. dm.) at ordinary temperature, and potential drop of about 0.15 volt between electrodes. With no addition agent present, the deposit formed as a mass of loosely adherent, sparkling, dendritic needles, which soon short-circuited the electrodes. Eight addition agents were tried: tannin, gum arabic, resorcin, gelatine, glucose, grape sugar, saccharine and glycerine, which were added as aqueous solutions. Tannin was found to be the best addition agent for this solution: 1 g. per 1,500 c.c. electrolyte caused the cathode deposit to form as an adherent, dense, fine, crystalline mass. Gum arabic and resorcin were less effective than tannin. Gelatine, glucose, grape-sugar, saccharine and glycerine did not have much effect.

*c*. Sodium-stannous fluoride electrolyte contained 8 g. of tin and 5 g. of sodium as fluorides. The electrolysis was conducted with current density of 10 amp. per sq. ft. (1.1 amp. per sq. dm.), and potential drop of about 0.23 volt between electrodes, at ordinary temperature. The same additions were tried, as those used for the stannous fluoride electrolytes (*b*). Tannin was most

satisfactory when added in proportion of 1 g. per 2,000 c.c. of electrolyte, which caused the cathode deposit to form as a smooth, crystalline and firmly adherent coating. Gelatine in proportion of 1 g. per 1,000 c.c. electrolyte almost as effective as tannin. Gum arabic in proportion of 1 g. per 1,500 c.c. electrolyte and resorcin in proportion of 1 g. per 750 c.c. electrolyte caused the tin to deposit as adherent small crystals. Glucose, grape-sugar, saccharine and glycerine had very little effect upon the deposition of the cathode tin.

16. Mathers and Cockrum ran a series of experiments on the effect of the presence of organic addition agents in electrolytes of tin salts of mineral acids. The research involved 179 various combinations of conditions and compositions of electrolytes, but in no case was an entirely satisfactory cathode deposit produced. The baths contained 4 g. of tin per 100 c.c. in solution as stannous chloride, stannous fluoride, stannous perchlorate, stannous fluoborate, and stannic chloride, and about 1 g. of free acid per 100 c.c. The addition agents tested were: Clove oil, peptone, gelatine, glue, phloridizin, Barbadoes aloes, sandalwood oil, residue from Curacao aloes, tannic acid, levulose, eucalyptus oil, catechu, benzoic acid, caffeine, pyrogallol, formin, licorice extract, oxalic acid, salicin, saponin, podophyllin, jalapin, digitalin, morphine, and gum arabic; 0.05 to 0.5 percent of each of these was added to the respective electrolytes, and their effect upon the cathode deposit noted. The current density was 0.4 amp. per sq. dm (3.72 amp. per sq. ft.), and ordinary temperature. Temperature of electrolyte at 80° C. did not materially improve the deposits. The best deposits were obtained at room temperature from stannous fluoborate electrolyte saturated with clove oil, or containing 0.1 percent of digitalin, and from stannous perchlorate electrolyte containing 0.1 percent phloridizin. These deposits were firm and free from loose trees or projections, but they were very crystalline. The authors stated that no combination of the salts, addition agents and conditions that were tried in their work will give a good tin-plating or refining-bath. (Trans. Amer. Electrochem. Soc. (1914), **26**, 133.)

17. Kern and Chen conducted experiments on the electrolytic refining of impure tin bullion, using electrolytes of stannous chloride, magnesium-stannous chloride, sodium-stannous fluoride,

stannous fluosilicate, and a mixture of stannous fluoride and stannous fluosilicate. Each of these solutions was prepared to contain 5 g. of tin per 100 c.c. The anodes contained 96 percent tin, 0.5 percent iron, 0.1 percent arsenic, 0.2 percent antimony, 2.0 percent lead, 0.6 percent copper, 0.1 percent bismuth, and 0.5 percent silver. The cathodes were sheets of pure tin. The electrolysis was conducted with current density of 12 and 24 amp. per sq. ft. (1.3 and 2.6 amp. per sq. dm.) and at 20° C. Distance between electrodes was 1.75 inches (4.4 cm.).

With no addition agents present the best cathode deposits were formed from the sodium-stannous chloride and magnesium-stannous chloride electrolytes; the cathode deposits formed from ammonium-stannous chloride were not quite so good, but were better than those formed from electrolytes of stannous chloride, sodium-stannous fluoride, and stannous fluosilicate.

The electrolytes which were selected to be tried with presence of addition agents were: Stannous chloride containing 5 g. of tin and 2.5 g. of free hydrochloric acid per 100 c.c.; sodium-stannous chloride containing 5 g. of tin as $SnCl_2.2NaCl$ and 2.5 g. of excess NaCl per 100 c.c.; ammonium-stannous chloride containing 5 g. of tin as $SnCl_2.2NH_4Cl$ per 100 c.c.; stannous fluosilicate containing 5 g. of tin as $SnSiF_6$ and 7.5 g. of free $H_2SiF_6$ per 100 c.c.; and stannous fluosilicate containing 5 g. of tin as $SnSiF_6$, 6 g. free $H_2SiF_6$, and 4 g. free HF per 100 c.c. The addition agents tried were aloin, gelatine, peptone (from meat), quinoline, clove oil, and boric acid; water was used as solvent for aloin, gelatine and peptone; alcohol was used for making solutions of quinoline and clove oil; boric acid was added dry. The effect of the addition agents was to restrain the formation of non-adherent cathode deposits of needles, spangles and dendrites.

Stannous fluosilicate electrolytes were found to be the best. The presence of free $H_2SiF_6$ and free HF was found to be beneficial: 1 g. aloin per 500 c.c. of electrolyte caused best cathode deposits to form; 1 g. gelatine per 500 c.c. of electrolyte caused formation of satisfactory deposits; 1 g. peptone per 1,000 c.c. electrolyte also produced satisfactory deposits.

The deposits of tin formed in sodium-stannous chloride, ammonium-stannous chloride, and stannous chloride electrolytes were

all similar; 1 g. peptone per 500 c.c. of each of these electrolytes caused formation of satisfactory cathode deposits.

The potential drop between the anodes and cathodes, when 1.25 inch apart, ranged from 0.1 to 0.2 volt in all cases. The presence of the addition agents which proved effective in restraining formation of non-adherent crystals in the cathode caused a rise in the potential drop of approximately 0.01 to 0.03 volt. (Trans. Amer. Electrochem Soc. (1918), **33**, 155-168.)

## EPITOME.

Conditions which are necessary for the electrolytic refining of tin and for the formation of solid adherent cathode deposits are:

(*a*) Electrolyte made of a tin salt which is very soluble; in most cases the presence of free acid is beneficial.

(*b*) Pure electrolyte with respect to metals which stand below tin in the E.M.F. and solution-pressure series. Electrolyte must not be too acid nor too alkaline.

(*c*) Not too low tin content of electrolyte with relation to current density; the higher the current density used the higher must be the tin content of the electrolyte.

(*d*) Circulation of the electrolyte at such a rate that segregation and polarization are prevented.

(*e*) Warm electrolytes produce, as a rule, the most satisfactory cathode deposits, and also cause more even corrosion of the anode. The potential drop in warm electrolytes is lower than in cold electrolytes.

(*f*) Presence of suitable addition agents prevents the tin depositing on the cathode as non-adherent crystals. The effect of the addition agent, when properly selected, is to cause the cathode deposit to be smoother, denser, more adherent, and purer; but when not properly selected the opposite effect results.

## EXPERIMENTAL PART.

The experiments and tests which are reported in Tables I to VIII, inclusive, were made by Mr. B. C. Tsen, in the Electrometallurgical Laboratory of the School of Mines of Columbia University. The electrolytes which were selected for trial are the following:

(*A*) Stannous-sodium chloride electrolyte, which contained 6 g. of tin as $SnCl_2.2NaCl$ and 2.5 g. of sodium chloride in excess per 100 c.c. This solution was prepared by taking calculated amounts of stannous chloride and sodium chloride, dissolving them in the required quantity of water, and filtering.

(*B*) Stannous fluoborate electrolyte, which contained 6 g. of tin as $Sn(BF_4)_2$ and 5 g. free $HBF_4$ per 100 c.c. It was prepared by taking a calculated amount of Baker's hydrofluoric acid (containing 48 g. HF per 100 c.c.), placing it in a copper vessel, and slowly adding a calculated amount of boric acid, according to $4HF + H_3BO_3 = HBF_4 + 3H_2O$. The reaction liberates considerable heat, so care was required in preparing the fluoboric acid, in order to prevent loss of the $HBF_4$ by volatilization, and also to prevent personal injury by the hydrofluoric acid vapors. After the fluoboric acid had cooled, a calculated amount of copper carbonate (equivalent to the amount of tin required) was added, forming copper fluoborate. The copper was then precipitated by means of granulated pure tin forming stannous fluoborate. The solution was diluted to contain the required amount of tin, and the acid content adjusted by addition of fluoboric acid.

(*C*) Stannous naphthalene-sulphonate electrolyte, which contained 6 g. tin and about 10 g. of free naphthalene-sulphonic acid per 100 c.c. The solution was prepared by placing a weighed amount of naphthalene in a flask and adding 10 percent excess of sulphuric acid (sp. gr. 1.84), according to:

$$C_{10}H_8 + H_2SO_4 = (C_{10}H_7)SO_3.H + H_2O.$$

The mixture was kept at 90° to 95° C. for 10 hours, allowed to cool, then poured into three volumes of cold water. Filtered off the remaining naphthalene crystals, leaving a solution of naphthalene-sulphonic acid containing a small amount of sulphuric acid. The tin naphthalene-sulphonate electrolytes were prepared in two ways: One way was by electrolyzing the solution, using pure tin anodes and cathodes, producing solution *Ca*. The cathode area was one-third that of the anode, which with 6 amp. per sq. ft. (0.66 amp. per sq. dm.) of anode gave 18 amp. per sq. ft. (2 amp. per sq. dm.) of cathode. Under this condition tin was dissolved from the anodes more rapidly than it was deposited on the cathodes, as with the higher cathode current density a large

amount of hydrogen was also liberated. After about 36 hours' continuous electrolysis a very small amount of hydrogen was liberated. The tin was deposited as non-adherent crystals, and periodically removed from the cell so as to prevent short-circuiting the electrodes. The solution was filtered before using for the tests. The better and more rapid way of preparing tin naphthalene-sulphonate electrolyte was by adding a calculated amount of copper carbonate to the naphthalene-sulphonate solution, forming copper naphthalene-sulphonate ($Cu(SO_3.C_{10}H_7)_2$). The copper was then replaced by tin by adding granulated pure metallic tin to the solution, completely precipitating the copper and forming tin naphthalene-sulphonate ($Sn(SO_3.C_{10}H_7)_2$); this solution is designated as solution *Cb*.

(*D*) Three stannous sulphate electrolytes containing 6 g. of tin, and 2.5 g., 5 g. and 7 g. free sulphuric acid per 100 c.c. These three solutions, designated as solutions *Da, Db* and *Dc,* were prepared by dissolving a calculated amount of copper sulphate in the required volume of water, adding the respective amounts of sulphuric acid, and then replacing the copper by means of granulated pure metallic tin. The copper was completely precipitated, giving pure solutions of stannous sulphate. The three solutions were filtered and used for the tests.

The anodes contained 95 percent tin, 3 percent bismuth, 0.5 percent lead, 0.5 percent antimony, and 0.5 percent silver; size 2.25 inches (5.7 cm.) wide, 3.5 inches (9 cm.) long, and 0.25 inch (0.6 cm.) thick. The cathodes were sheets of pure tin made by pouring the molten metal over the surface of an iron plate set at an angle of about 20 degrees. The cathodes were about 1/16 inch (0.16 cm.) thick and of approximately same width and length as the anodes. About 300 c.c. of electrolyte was used for each test and the electrolysis conducted with current density of 16 amp. per sq. ft. (1.8 amp. per sq. dm.). Distance between electrodes 1.75 inches (4.4 cm.). Temperatures of electrolyte were 20° C. and 40° C.

The addition agents tried were peptone (from meat), gelatine, aloin and engine-oil. The peptone, gelatine and aloin were added as aqueous solutions; and the engine-oil as an emulsion prepared by bubbling steam into a small amount of the oil, the excess oil

was removed and the white emulsion used. The results of the test are given in Tables I to VIII, inclusive.

In order to determine whether tin would be dissolved when the electrodes were allowed to remain in sulphate electrolytes and when no current is passing a strip of tin weighing 22.91 g. was suspended for 24 hours in sulphuric acid solution containing

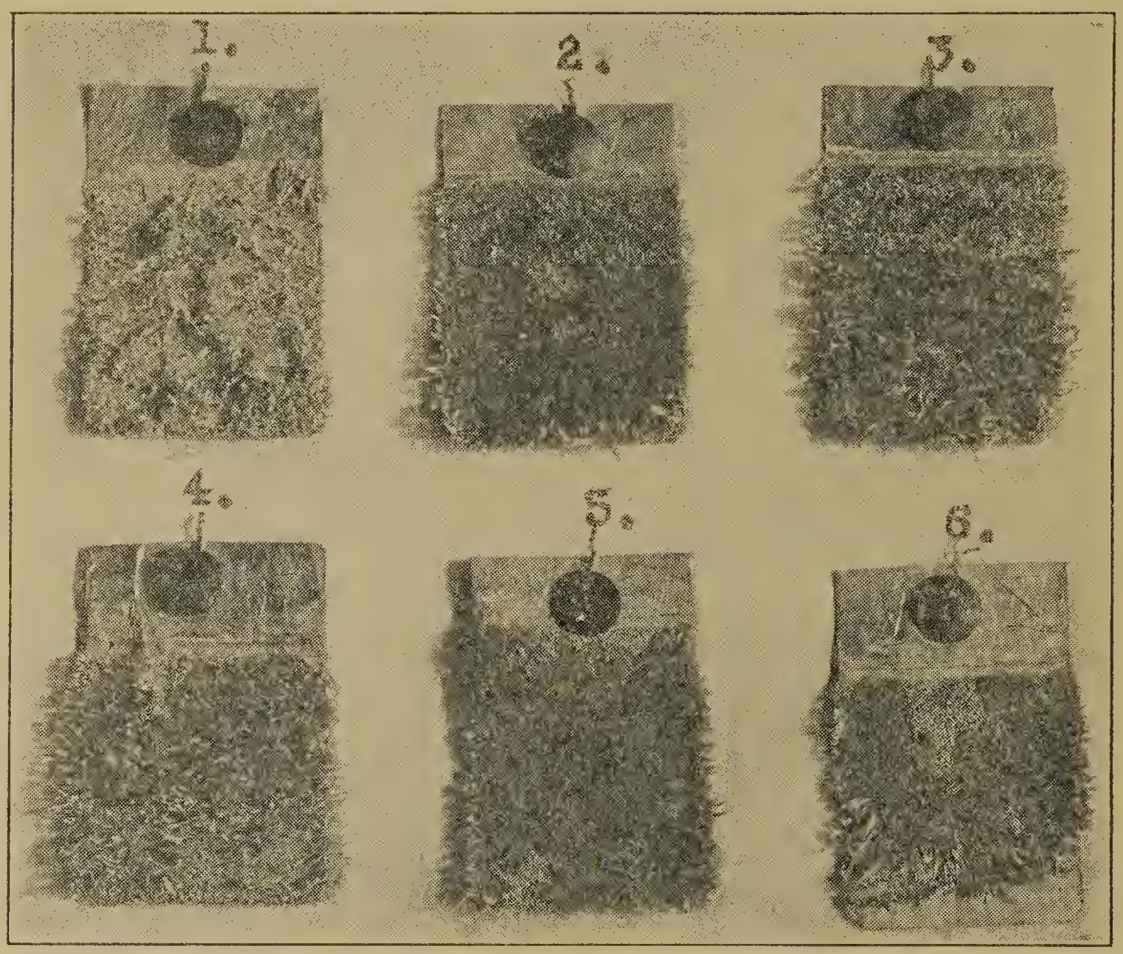


FIG. 1. Cathodes 1, 2, 3, 4, 5, and 6 were produced in electrolytes containing 1 g. gelatine and 8 g. oil emulsion per 1000 c.c.

| CATHODES. | SOLUTIONS IN WHICH PRODUCED. | CATHODE WHICH REPORTED IN: |
|---|---|---|
| 1. | Sodium-stannous chloride (*A.*) | Table II as Test No. 12. |
| 2. | Stannous fluoborate (*B.*) | " III " " " 12. |
| 3. | Naphthalene-sulphonate (*Cb.*) | " IV " " " 12. |
| 4. | Stannous sulphate (*Da.*) | " V " " " 12. |
| 5. | " " (*Db.*) | " VI " " " 12. |
| 6. | " " (*Dc.*) | " VII " " " 12. |

approximately 9 g. $H_2SO_4$ per 100 c.c. The loss in weight was 0.04 g., showing that tin is only slightly attacked by dilute sulphuric acid. Former experiments showed that fluoboric acid solutions do not dissolve metallic tin.

The photographs show the kinds of deposits which are formed in the different solutions. These are of cathodes which were in electrolytes containing 1 g. of peptone plus 8 g. oil emulsion per

1,000 c.c. solution, and 1 g. gelatine plus 8 g. oil emulsion per 1,000 c.c. solution. Temperature of electrolytes was 40° C.

Several current efficiency tests were made so as to show the effect of the formation of solid cathode deposits upon the current efficiency; or else the effect of the addition agent and of the electrolyte upon the current efficiency. Low current efficiencies result

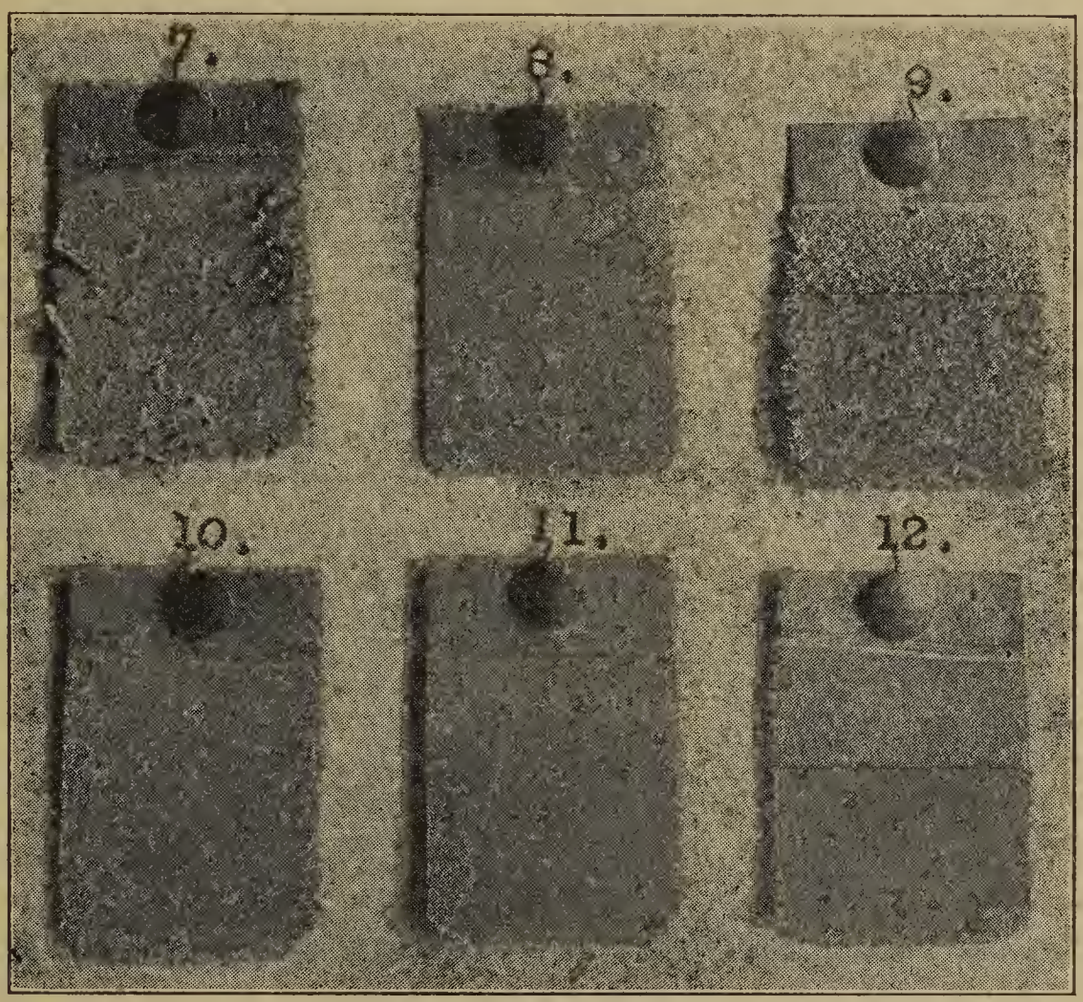


FIG. 2. Cathodes 7, 8, 9, 10, 11 and 12 were produced in electrolytes containing 1 g. peptone and 8 g. oil emulsion per 1000 c.c.

| CATHODES. | SOLUTIONS IN WHICH PRODUCED. | CATHODE WHICH REPORTED IN: |
|---|---|---|
| 7. | Sodium-stannous chloride (*A.*) | Table II as Test No. 11. |
| 8. | Stannous fluoborate (*B.*) | " III " " " 11. |
| 9. | Naphthalene-sulphonate (*Cb.*) | " IV " " " 11. |
| 10. | Stannous sulphate (*Da.*) | " V " " " 11. |
| 11. | " " (*Db.*) | " VI " " " 11. |
| 12. | " " (*Dc.*) | " VII " " " 11. |

when rough, non-adherent or long, crystalline deposits are formed. The higher current efficiencies are when the smoothest, most dense, and most adherent cathode deposits are formed (see Table VIII). A copper coulometer was in circuit with the cells containing the tin electrolytes. The values used for making the calculations were 1.1858 g. copper and 2.2142 g. tin per ampere-hour.

## SUMMARY OF RESULTS.

*No Addition Agents Present:* The best cathode deposits which were formed in electrolytes, which contained no addition agents, were those from stannous sulphate solution containing 6 g. tin and 7 g. of sulphuric acid per 100 c.c. (Solution *Dc*). The deposits formed in stannous naphthalene-sulphonate electrolytes (Solution *Cb*), which was made by replacing copper by tin, were second best; and the deposits from stannous-sodium chloride electrolyte (Solution *A*) and from stannous fluoborate electrolyte (Solution *B*) were third best. In no case were adherent, smooth cathode deposits formed, with no addition agent present.

All of the electrolytes except stannous sulphate containing 2.5 g. free sulphuric acid per 100 c.c. (Solution *Da*) remained clear on standing: this solution became turbid, and after several days a white precipitate settled out.

As the cathode deposits formed from stannous naphthalene-sulphonate electrolyte, solution *Ca* (which was prepared by electrolysis), were less satisfactory than those formed from the stannous naphthalene-sulphonate electrolyte, solution *Cb* (which was made from the corresponding copper solution by replacement with tin), solution *Ca* was omitted from the tests which were run with the presence of addition agents.

*Addition Agents Present:* In order to produce satisfactory cathode deposits of tin in the electrolytic refining of tin, it seems to be necessary to add suitable addition agents to the electrolyte. In the tests which are reported in Tables I to VIII, inclusive, it was found that the presence of peptone and of gelatine, were beneficial; the presence of aloin was beneficial only with the fluoborate electrolytes when present in proportion of 1 g. per 1,000 c.c. solution. The most satisfactory cathode deposits were formed in the stannous fluoborate and in the stannous sulphate electrolytes when gelatine or peptone were present in the proportion of 1 g. per 500 to 1,000 c.c. of electrolyte. In the case of the sulphate electrolyte, the best cathode deposits were formed when the solutions contained 5 or 7 g. free $H_2SO_4$ per 100 c.c.; more than 2.5 g. free $H_2SO_4$ per 100 c.c. solution seems to be necessary. The presence of free acid over 2.5 g. per 100 c.c. keeps the solution clear, and prevents the precipitation of basic salts, which if allowed to collect on the cathode deposit causes the tin to form

non-adherent and as large crystals. The cathode deposits formed from the stannous sulphate electrolyte containing 5 and 7 g. free $H_2SO_4$ per 100 c.c. were as satisfactory as those formed from stannous fluoborate electrolyte.

When dilute aqueous solutions of addition agents were added to the electrolytes, slight turbidity resulted, with exception of the fluoborate and the naphthalene-sulphonate electrolytes. The greatest turbidity formed in the chloride electrolyte and in the sulphate electrolyte containing 2.5 g. free $H_2SO_4$ per 100 c.c.

Heating the electrolytes is beneficial in that the potential drop between the electrodes is reduced, and also a more adherent deposit is formed. Raising the temperature increases the effectiveness of the addition agent; a smaller amount of addition agent being required.

This work will be continued by testing the effect of other addition agents and combinations of two or more addition agents in the electrolytes which are reported in this article.

TABLE I.

*No Addition Agent Present.*

C. D. = 16 amp. per sq. ft. (1.8 amp. per sq. dm.).
Temperature 20° to 23° C.

| Solution | Composition of Electrolyte. 6 g. Tin + the following per 100 cc. Solution | Average Potential Drop. Volt | Character of Cathode Deposits |
|---|---|---|---|
| *A* | $SnCl_2.2NaCl$ + 2.5 g. NaCl | 0.42 | Mass of crystals over surface, and bright moss-like dendrites along edges. |
| *B* | $Sn(BF_4)_2$ + 5 g. $HBF_4$ | 0.31 | Mass of bright needles with dendrites along edges. |
| *Ca* | $Sn(SO_3.C_{10}H_7)_2$ + Free Acid made by electrolysis | 0.28 | Mass of bright crystals at start, and after two hours amorphous, gray deposit formed. |
| *Cb* | $Sn(SO_3.C_{10}H_7)_2$ + 10 g. $H.SO_3.C_{10}H_7$. Made from copper solution | 0.22 | Adherent, even mass of bright needle crystals, dendrites along edges. |
| *Da* | $SnSO_4$ + 2.5 g. $H_2SO_4$ | 0.25 | Mass of bright needles with long dendrites along the edges. |
| *Db* | $SnSO_4$ + 5 g. $H_2SO_4$ | 0.22 | Mass of short needle crystals, and small dendrites along the edges. |
| *Dc* | $SnSO_4$ + 7 g. $H_2SO_4$ | 0.22 | Mass of even, small needles, and short dendrites along the edges. Best deposit of this series. |

TABLE II.

*Stannous Sodium Chloride Electrolytes Containing 6 Grams Tin and 2.5 Grams Excess NaCl per 100 c.c. (Solution A.)*

Current Density = 16 amp. per sq. ft. (1.8 amp. per sq. dm.)

| Test No. | Addition Agent Present | | Average Potential Drop. Volt | Temperature ° C. | Character of Cathode Deposits |
|---|---|---|---|---|---|
| | Name | 1 gr. per c.c. Electrolyte | | | |
| 1 | Peptone | 1,000 | 0.46 | 20-23 | Dense, crystalline, with dendrites along edges. |
| 2 | " " | 500 | 0.56 | 20-23 | Dense, crystalline, with long needles and dendrites along edges. |
| 3 | " " | 250 | 0.42 | 20-23 | Dense mass of gray smooth adherent crystals, with few larger crystals along edges. Fairly good. |
| 4 | Gelatine | 1,000 | 0.43 | 20-23 | Crystalline deposit, with long dendrites along edges. |
| 5 | " " | 500 | 0.48 | 20-23 | Similar deposit to No. 4. |
| 6 | " " | 250 | 0.50 | 20-23 | Deposit poorer than Nos. 4 and 5. |
| 7 | Aloin | 1,000 | 0.42 | 20-23 | Mass of needles and dendrites. |
| 8 | " | 500 | 0.44 | 20-23 | Adherent, crystalline deposit. Fairly good. |
| 9 | " | 250 | 0.42 | 20-23 | Mass of non-adherent moss-like dendritic crystals, which soon short-circuited electrodes. |
| 10 | " | 500 | 0.18 | 40 | Adherent, dense, crystalline, with heavy long dendrites along edges. |
| 11 | Peptone and Oil Emulsion | 1,000<br>125 | 0.16 | 40 | Mass of crystals, with long dendrites along edge, which soon short-circuited electrodes. |
| 12 | Gelatine and Oil Emulsion | 1,000<br>125 | 0.24 | 40 | Mass of intermixed long crystals and dendrites. |
| 13 | Gelatine | 250 | 0.22 | 40 | Mass of adherent crystals, with dendrites along edges, which soon short-circuited electrodes. |

## Table III.

*Stannous Fluoborate Electrolytes Containing 6 Grams Tin and 5 Grams Free $HBF_4$ per 100 c.c. (Solution B.)*

Current Density = 16 amp. per sq. ft. (1.8 amp. per sq. dm.)

| Test No. | Addition Agent Present | | Average Potential Drop. Volt | Temperature ° C. | Character of Cathode Deposits |
|---|---|---|---|---|---|
| | Name | 1 gr. per c.c. Electrolyte | | | |
| 1 | Peptone | 1,000 | 0.44 | 20-23 | Adherent, dense, crystalline. Fairly good. |
| 2 | " " | 500 | 0.44 | 20-23 | Adherent, very dense, crystalline deposit. Good. |
| 3 | " " | 250 | 0.52 | 20-23 | Similar to No. 2 deposit. Good. |
| 4 | Gelatine | 1,000 | 0.26 | 20-23 | Adherent, smooth, dense deposit. Very good. |
| 5 | " " | 500 | 0.30 | 20-23 | Similar to No. 4 deposit. Very good. |
| 6 | " " | 250 | 0.34 | 20-23 | Similar to Nos. 4 and 5 deposits. Very good. |
| 7 | Aloin | 1,000 | 0.30 | 20-23 | Adherent, very smooth, dense deposit. Very good. |
| 8 | " | 500 | 0.34 | 20-23 | Adherent mass of large crystals, with needles along edges. |
| 9 | " | 250 | 0.40 | 20-23 | Adherent, dense, smooth, crystalline deposit. Good. |
| 10 | " | 500 | 0.28 | 40 | Adherent, dense, crystalline deposit, with few short needles along edge. Fairly good. |
| 11 | Peptone and Oil Emulsion | 1,000<br>125 | 0.18 | 40 | Adherent, dense, smooth, finely crystalline deposit. Very good. |
| 12 | Gelatine and Oil Emulsion | 1,000<br>125 | 0.16 | 40 | Mass of gray fine crystals, with needles along edges. |
| 13 | Gelatine | 250 | 0.20 | 40 | Adherent, dense mass of crystals, with some short needles along edges. Fairly good. |

## Table IV.

*Stannous Naphthalene-Sulphonate Electrolytes Containing 6 Grams Tin, 10 Grams Free $H.SO_3.C_{10}H_7$ and Small Amount $H_2SO_4$ per 100 c.c. (Solution Cb.)*

Current Density = 16 amp. per sq. ft. (1.8 amp. per sq. dm.)

| Test No. | Addition Agent Present | | Average Potential Drop. Volt | Temperature ° C. | Character of Cathode Deposits |
|---|---|---|---|---|---|
| | Name | 1 gr. per c.c. Electrolyte | | | |
| 1 | Peptone | 1,000 | 0.24 | 20-23 | Mass of dense, small crystals covering entire cathode. |
| 2 | " " | 500 | 0.24 | 20-23 | Adherent, even mass of bright needle crystals. |
| 3 | " " | 250 | 0.22 | 20-23 | Mass of dense, short, needle crystals, longer on edges of cathode. |
| 4 | Gelatine | 1,000 | 0.32 | 20-23 | Moderately adherent, smooth, amorphous deposit. |
| 5 | " " | 500 | 0.34 | 20-23 | Grayish-white, heavy needles, not so good as when no gelatine present. |
| 6 | " " | 250 | 0.36 | 20-23 | Similar to No. 5. |
| 7 | Aloin | 1,000 | 0.32 | 20-23 | Non-adherent dendrites. |
| 8 | " | 500 | 0.36 | 20-23 | Non-adherent dendrites; smaller crystals than No. 7. |
| 9 | " | 250 | 0.36 | 20-23 | Non-adherent dendrites, similar to No. 7. |
| 10 | " | 500 | 0.24 | 40 | Mass of adherent, small, bright crystals for 2 hours, after which long needles continued to form. |
| 11 | Peptone and Oil Emulsion | 1,000<br>125 | 0.22 | 40 | Mass of heavy needles intermixed with dendrites. |
| 12 | Gelatine and Oil Emulsion | 1,000<br>125 | 0.24 | 40 | Non-adherent, long needles. |
| 13 | Gelatine | 250 | 0.28 | 40 | Loose, long needles, which soon short-circuited electrodes. |

## Table V.

*Stannous Sulphate Electrolytes Containing 6 Grams Tin and 2.5 Grams Free $H_2SO_4$ per 100 c.c. (Solution Da.)*

Current Density = 16 amp. per sq. ft. (1.8 amp. per sq. dm.)

| Test No. | Addition Agent Present | | Average Potential Drop. Volt | Temperature ° C. | Character of Cathode Deposits |
|---|---|---|---|---|---|
| | Name | 1 gr. per c.c. Electrolyte | | | |
| 1 | Peptone | 1,000 | 0.36 | 20-23 | Adherent, dense, even bright, finely crystalline deposit. Fair. |
| 2 | " " | 500 | 0.35 | 20-23 | Adherent, dense, gray, finely crystalline, with few larger crystals along edges. |
| 3 | " " | 250 | 0.34 | 20-23 | Adherent, smooth, grayish, finely crystalline deposit. Good. |
| 4 | Gelatine | 1,000 | 0.22 | 20-23 | Adherent, smooth, bright, finely crystalline deposit. Good. |
| 5 | " " | 500 | 0.20 | 20-23 | Adherent mass of rough, grayish, dense, fine crystals. Fair. |
| 6 | " " | 250 | 0.24 | 20-23 | Adherent, smooth, bright, finely crystalline deposit. Good. |
| 7 | Aloin | 1,000 | 0.22 | 20-23 | Adherent, dense, smooth, grayish, finely crystalline deposit. Good. |
| 8 | " | 500 | 0.22 | 20-23 | Adherent, dense, smooth, finely crystalline deposit, with few short needles. Fair. |
| 9 | " | 250 | 0.24 | 20-23 | Adherent, dense, bright, smooth, finely crystalline deposit. Very good. |
| 10 | " | 500 | 0.16 | 40 | Adherent, crystalline deposit, intermixed with small needles, and longer needles along edges. |
| 11 | Peptone and Oil Emulsion | 1,000<br>125 | 0.22 | 40 | Adherent, dense, smooth, finely crystalline deposit. Good. |
| 12 | Gelatine and Oil Emulsion | 1,000<br>125 | 0.24 | 40 | Non-adherent, bright, long needles. |
| 13 | Gelatine | 250 | 0.33 | 40 | Adherent, dense, even, finely crystalline deposit, some needles and small dendrites along edges. Fair. |

## Table VI.

*Stannous Sulphate Electrolytes Containing 6 Grams Tin and 5 Grams Free $H_2SO_4$ per 100 c.c. (Solution Db.)*

Current Density = 16 amp. per sq. ft. (1.8 amp. per sq. dm.)

| Test No. | Addition Agent Present | | Average Potential Drop. Volt | Temperature ° C. | Character of Cathode Deposits |
|---|---|---|---|---|---|
| | Name | 1 gr. per c.c. Electrolyte | | | |
| 1 | Peptone | 1,000 | 0.28 | 20-23 | Adherent, dense, smooth, grayish, finely crystalline deposit. Good. |
| 2 | " " | 500 | 0.28 | 20-23 | Adherent, dense, smooth, grayish, finely crystalline deposit, with few large crystals along the edges. Fair. |
| 3 | " " | 250 | 0.24 | 20-23 | Adherent, dense, smooth, grayish, finely crystalline deposit. Good. |
| 4 | Gelatine | 1,000 | 0.20 | 20-23 | Adherent, dense, smooth, bright, finely crystalline deposit. Good. |
| 5 | " " | 500 | 0.18 | 20-23 | Adherent, dense, smooth, bright, finely crystalline deposit. Good. |
| 6 | " " | 250 | 0.18 | 20-23 | Adherent, dense, smooth deposit of fine crystals, with some small needles along edges. Fair. |
| 7 | Aloin | 1,000 | 0.18 | 20-23 | Adherent, dense, smooth, finely crystalline, with few short needles along edges. Fair. |
| 8 | " | 500 | 0.18 | 20-23 | Similar to No. 7. Fair. |
| 9 | " | 250 | 0.20 | 20-23 | Slightly better than Nos. 7 and 8. |
| 10 | " | 500 | 0.12 | 40 | Smooth, dense, bright, finely crystalline deposit, with slender dendrites along edges. |
| 11 | Peptone and Oil Emulsion | 1,000<br>125 | 0.21 | 40 | Adherent, smooth, dense, finely crystalline deposit. Very good. |
| 12 | Gelatine and Oil Emulsion | 1,000<br>125 | 0.22 | 40 | Adherent, dense deposit of mixture of very small needles and fine crystals, with short needles along edges. Fair. |
| 13 | Gelatine | 250 | 0.14 | 40 | Adherent, dense, finely crystalline deposit, with needles and dendrites along edges. Fair. |

## Table VII.

*Stannous Sulphate Electrolytes Containing 6 Grams Tin and 7 Grams Free $H_2SO_4$ per 100 c.c. (Solution Dc.)*

Current Density = 16 amp. per sq. ft. (1.8 amp. per sq. dm.)

| Test No. | Addition Agent Present | | Average Potential Drop. Volt | Temperature ° C. | Character of Cathode Deposits |
|---|---|---|---|---|---|
| | Name | 1 gr. per c.c. Electrolyte | | | |
| 1 | Peptone | 1,000 | 0.28 | 20-23 | Adherent, very dense, smooth, bright, finely crystalline deposit. Very good. |
| 2 | " " | 500 | 0.24 | 20-23 | Adherent, dense, smooth, bright, finely crystalline deposit. Good. |
| 3 | " " | 250 | 0.24 | 20-23 | Similar to No. 1. Very good. |
| 4 | Gelatine | 1,000 | 0.18 | 20-23 | Adherent, dense, smooth, grayish, finely crystalline deposit. Good. |
| 5 | " " | 500 | 0.18 | 20-23 | Similar to No. 4. Good. |
| 6 | " " | 250 | 0.18 | 20-23 | Similar to Nos. 4 and 5. Good. |
| 7 | Aloin | 1,000 | 0.18 | 20-23 | Moderately adherent mass of bright, fine crystals. |
| 8 | " | 500 | 0.18 | 20-23 | Moderately adherent mass of grayish, fine crystals, with needles along edges. |
| 9 | " | 250 | 0.26 | 20-23 | Adherent, smooth, crystalline deposit. Fair. |
| 10 | " | 500 | 0.23 | 40 | Mass of heavy, short needles. |
| 11 | Peptone and Oil Emulsion | 1,000<br>125 | 0.23 | 40 | Adherent, smooth, dense, bright, finely crystalline deposit. Very good. |
| 12 | Gelatine and Oil Emulsion | 1,000<br>125 | 0.25 | 40 | Non-adherent mass of short needles and spangles, with long needles and spangles along edges. |
| 13 | Gelatine | 250 | 0.32 | 40 | Loose, long needles, which soon short-circuited electrodes. |

## Table VIII.

*Current Efficiency. Time: 5 Hours.*

Current Density = 16 amp. per sq. ft. (1.8 amp. per sq. dm.) Temp. 40°C.

| No. Run | Electrolyte Containing 6 g. Tin per 100 c.c. | Addition Agent Present | | Current Efficiency in Percent | Character of Cathode Deposits |
|---|---|---|---|---|---|
| | | Name | 1 g. per c.c. Electrolyte | | |
| 1 | $SnCl_2.2NaCl$ + 2.5 gm. NaCl (Solution *A*) | Peptone..... | 1,000 | 49 | Crystals and dendrites. |
| | | Oil Emulsion | 125 | | |
| 2 | $Sn(BF_4)_2$ + 5 gm. $HBF_4$ (Solution *B*) | Peptone..... | 1,000 | 80 | Crystalline deposit, with needles along edges. |
| | | Oil Emulsion | 125 | | |
| 3 | $Sn(SO_3.C_{10}H_7)_2$ (Solution *Cb*) | Peptone..... | 1,000 | 80 | Heavy, long needles. |
| | | Oil Emulsion | 125 | | |
| 4 | $SnSO_4$ + 2.5 gm. $H_2SO_4$ (Solution *Da*) | Peptone..... | 1,000 | 81 | Mass of long needles. |
| | | Oil Emulsion | 125 | | |
| 5 | $SnSO_4$ + 5 gm. $H_2SO_4$ (Solution *Db*) | Peptone..... | 1,000 | 80 | Adherent crystals, with short needles along edges. |
| | | Oil Emulsion | 125 | | |
| 6 | $SnSO_4$ + 7 gm. $H_2SO_4$ (Solution *Dc*) | Peptone..... | 1,000 | 80 | Short needles and spangles, with larger dendrites along edges. |
| | | Oil Emulsion | 125 | | |
| 7 | $SnCl_2.2NaCl$ + 2.5 gm. NaCl (Solution *A*) | Gelatine..... | 250 | 52 | Mass of dense crystals, with dendrites along edges. |
| 8 | $Sn(BF_4)_2$ + 5 gm. $HBF_4$ (Solution *B*) | Gelatine..... | 250 | 100 | Dense, crystalline, with few short needles along edges. |
| 9 | $Sn(SO_3.C_{10}H_7)_2$ (Solution *Cb*) | Gelatine..... | 250 | 89 | Long needles and dendrites. |
| 10 | $SnSO_4$ + 2.5 gm. $H_2SO_4$ (Solution *Da*) | Gelatine..... | 250 | 100 | Dense, even, finely crystalline, with needles and dendrites along edges. |
| 11 | $SnSO_4$ + 5 gm. $H_2SO_4$ (Solution *Db*) | Gelatine..... | 250 | 97 | Dense, crystalline, with needles and dendrites along edges. |
| 12 | $SnSO_4$ + 7 gm. $H_2SO_4$ (Solution *Dc*) | Gelatine..... | 250 | 99 | Dense, crystalline, with needles and dendrites along edges. |

*Electro-metallurgical Laboratory,*
*School of Mines of Columbia University,*
*New York City, August, 1920.*

## DISCUSSION.

W. S. LANDIS[1]: The electrolytic refining of tin is becoming of more and more commercial importance every day. I understand we have just lost our second largest copper refinery in New Jersey or New York, by its being converted into a tin refinery, so this paper should bring out much discussion.

F. C. MATHERS[2]: The pictures of some of these cathodes indicate that the deposits were really very satisfactory. I am sorry Dr. Kern did not send some actual samples of those cathodes, and I suggest that it might be a good plan for the Secretary to request authors to furnish or send samples of work, whenever it is possible, to these meetings. I would like very much to examine those cathodes. In connection with the deposition from chloride solution, you will notice on page 152 that those deposits look like doormats. That is a characteristic of deposits from chloride solution. I have never been able to get a non-crystalline or smooth deposit from chlorides. Dr. Bancroft keeps asking why, and I do not know, and he does not know and has no suggestion to make. Wherever Dr. Kern says that the edges of the cathodes show slight crystals, that means that the deposits would not be so good, because there will be a short circuit before he obtains a satisfactory thickness of deposits. All deposits look fairly good in the very beginning, but where you undertake to build thick ones from poor baths, you encounter difficulties. I have tried naphthalene, benzol, phenol sulphonate and all the rest of them, and have never found any particular advantage in them over the simple sulfate. At the beginning of the war, the imports of tin from the Straits were cut off and the importation from South America very much increased. Now it happens that the South American tin is naturally impure, containing lead, and could not be used directly on sheet for food containers. That brought about the necessity of electrolytic refining, and a plant was started by the American Smelting and Refining Company at Maurer, N. J., for refining tin under the Whitehead patent. I think they used a tin fluosilicate bath, with

[1] American Cyanamid Co., New York City.
[2] Associate Prof. of Chemistry, Univ. of Indiana, Bloomington, Ind.

glue as an addition agent. The deposits were very loosely adherent to the cathode. In a commercial way, for the last three years, they have been using addition agents which prevent this crystalline structure, and get a very satisfactory cathode. The patents on that process have been allowed but not issued yet. These samples of tin cathodes were deposited from the same electrolytes and with the same addition agents that are used at this plant. This is an especially smooth cathode. It takes careful work to get a cathode of this thickness and this extraordinary smoothness, but it is quite easy to build up a solid cathode of this thickness. The addition agents mentioned by Dr. Kern would probably be too expensive for commercial use. For instance, this meat peptone, which he uses in his best bath, would probably cost $1.50 a pound. The cheapest tin bath, leaving out the chloride, of course, would be the stannous sulphate. I found that the addition of fluoborate or fluosilicate to that bath improves the deposit somewhat, and I might say that a bath of stannous fluoborate or stannous fluosilicate gives a better deposit than stannous sulphate, but the improvement might not be worth the extra cost on the bath. The current yields are almost 100 percent if the cathode is so firm that none of the deposit drops off. The thing that decreases the cathode yield is short circuiting.

Edward F. Kern: I also presented a paper on the "Electrolytic Refining of Tin" at a previous meeting of the Society.[3] The electrolytes referred to in that paper were the chlorides, fluorides and fluosilicates of tin, and the double chlorides and fluorides of tin with sodium, magnesium and ammonium. Last year I was informed that at a certain Japanese tin refinery, the double chloride of tin and sodium electrolyte with the addition of peptone and glue, as published in Vol. XXXIII of our Transactions, was used with success, and that no great trouble was encountered from short-circuiting, as continual voltage inspection of the tanks was maintained. When short-circuits were discovered, the "bridging" crystals were knocked from the cathodes by means of a thin strip of wood. The Japanese tank inspectors no doubt were more watchful and alert than the inspectors in our American refineries, which may account for the good work

[3] Trans. Am. Electrochem. Soc. (1918), 33, 155-168.

done with the double chloride electrolyte. Professor Mathers refers to the tin refinery of the American Smelting and Refining Company at Maurer, N. J., saying that he thought tin fluosilicate electrolyte with glue addition was used in this plant. The use of tin fluosilicate for the refining of tin was patented by Mennicke, and the use of fluosilicate electrolyte with the addition of gelatine or glue for the purpose of increasing the density of the cathode deposit was patented by Hollis (Nos. 10 and 11 of review of literature). When tin refining by electrolysis was started at Maurer, N. J., the fluosilicate electrolyte was selected. As their anodes contained lead, it was found necessary to add some acid, or acid radical, with which lead combines and forms an insoluble precipitate, otherwise the lead would dissolve along with the tin from the anode and be deposited with the tin on the cathode. Tin and lead are practically in the same position in the E. M. F. or solution-pressure series. Mr. Whitehead, who had charge of the tin refinery at Maurer, added sulphuric acid to their electrolyte, and applied for patents for same. He later made patent application for the use of tin fluoborate electrolyte with addition of hydrofluoric acid, for the purpose of "sliming" lead in tin anodes, and the use of a suitable addition agent for the purpose of causing the tin to deposit smoothly on the cathode. The presence of the hydrofluoric acid in this electrolyte was for the purpose of precipitating the lead as insoluble lead fluoride. The beneficial effect of free hydrofluoric acid in tin fluosilicate electrolyte was published in the Transactions of this Society, Vol. XXXIII, and is given in reference No. 17 (Kern and Chen). I found the fluoborate electrolyte to be more satisfactory than the fluosilicate, as it is more stable and also produces better deposits. This is also true for lead. As fluoborate is more expensive, it did not replace fluosilicate for refining purposes. The electrolyte which was used at the Maurer plant until recently was the fluosilicate of the following composition: 4 to 5 grams of tin, 4.5 to 7.5 grams free $H_2SiF_6$ (10 to 13 grams total $H_2SiF_6$), 1 to 2.5 grams $H_2SO_4$, and 0.5 to 1.5 HF per 100 c. c. The addition agents used were glue, cresol and oil, added in the required amounts as determined by trial. The current density used was between 10 and 12 amperes per square

foot (110-132 A. per sq. m.). The anodes contained between 94 and 98 percent Sn, 0.5 to 1.3 Pb, 0.3 to 0.6 Cu, 0.5 to 3.5 Bi, 0.02 to 0.35 As, 0.1 to 0.25 Sb, and 5 to 9 ounces of silver per ton (0.017 to 0.03 percent), and 0.01 to 0.02 ounce of gold per ton (0.34 to 0.68 g. per metric ton). The cathodes contained from 99.2 to 99.9 percent Sn, 0.01 to 0.0015 As, 0.01 to 0.09 Sb, 0.02 to 0.05 Bi, and 0.015 to 0.03 Cu. After it was published that tin sulphate electrolytes produced as satisfactory results as tin fluosilicate electrolytes, the company had comparative tests of these two electrolytes made. The outcome was that their fluosilicate electrolyte has been replaced by the sulphate electrolyte of composition approximating solution Db., Tables I and VI, different addition agents, however, being used, which I believe to be bone glue and cresol-sulphonic acid. Professor Mathers stated that the samples of cathode tin which were shown by him were formed from an electrolyte of the same composition (fluosilicate), and with the same addition agents as are used at the Maurer plant. He should have let us know what addition agents were used. As to meat peptone costing $1.50 per pound, I do not recommend that this be used in commercial refineries; but when conducting the researches, the idea was to find some addition agent which would cause the formation of smooth, adherent, solid deposits on the cathode, and then afterwards seek some cheap chemicals or materials whose composition approximates that of the expensive addition agent. The use of bone glue, cresol and oil emulsion gives the cheap mixture which causes tin to deposit satisfactorily from fluosilicate, fluoborate and sulphate electrolytes. As a rule, the addition agents which produce satisfactory cathode deposits with one electrolyte do not do so when used with other electrolytes. The proper addition agent or mixture of addition agents must be found for each electrolyte. Due to the lack of information and theory on addition agents, the selection of the proper addition agent for a certain electrolyte cannot be made by merely knowing its composition and molecular arrangement. No doubt this will be possible in the future.

*A paper presented at the Thirty-eighth General Meeting of the American Electrochemical Society, in Cleveland, October 2, 1920, President Landis in the Chair.*

# THE ELECTRO-DEPOSITION OF BRASS FROM CYANIDE SOLUTIONS.[1]

By Alfred L. Ferguson[2] and Earl G. Sturdevant.

## Abstract.

The author reviews briefly the existent literature, and shows where it is contradictory. He discusses at length the theory of the co-deposition of two metals from solution, and then describes various tests devised to throw light on the fundamental principles involved. He studies particularly the current efficiencies at both electrodes, the effects of varying temperature, current density, and proportions of acid, alkaline and neutral ingredients in the solution. [J. W. R.]

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[1] The work herein reported is from a thesis presented by E. G. Sturdevant to the Faculty of the Graduate School of the University of Michigan in part fulfilment of the requirements for the Degree of Doctor of Philosophy. Manuscript received August 4, 1920.

[2] Dept. of Chemistry, University of Michigan.

*Introduction.*

A survey of the literature on the electro-deposition of brass shows that brass has been obtained from cyanide solutions which vary decidedly in ratio of copper to zinc, metal content, cyanide content, alkalinity, current density, and temperature. This fact is clearly evident from the table compiled by C. W. Bennett (Trans. Amer. Electrochem. Soc. (1913), **23**, 251), which is a summary of brass plating solutions that have been recommended. Calculations from the figures given in this table show that the composition of these solutions, expressed in grams per liter, varies from 0.76 to 47.77 g. of copper; 1.59 to 23.38 g. of zinc; 0.00 to 165.00 g. of potassium cyanide; 0.00 to 180.00 g. of alkaline substances; and 0.00 to 90.00 g. of slightly acid substances. The extreme variation in metal ratio is shown by solutions 1 and 12 in this table. In solution 1 the ratio is 10 parts of copper to 80 parts of zinc; in solution 12 the ratio is 10 parts of copper to 1 part of zinc.

The electro-deposition of brass has been investigated by F. Spitzer (Zeitsch. Electrochem. (1905), **11**, 172), S. Field (Trans. Faraday Soc. (1909), **9**, 172), A. W. Davison (Jour. Phys. Chem. (1914), **18**, 488), and A. Hönig (Zeitsch. Electrochem. (1916), **22**, 286). They worked under decidedly different experimental conditions as well as with solutions of widely different composition. These different experimental conditions may, to some extent at least, account for the poor agreement of their results. The lack of conformity is readily apparent from Table I.

TABLE I.

| Grams Copper per Liter | Grams Zinc per Liter | Ratio of Copper to Zinc | Percent Copper Deposited | Investigator |
|---|---|---|---|---|
| 6.35 | 6.53 | 0.97 | 73.0 | Spitzer |
| 3.17 | 9.85 | 0.32 | 70.0 | " |
| 34.25 | 22.00 | 1.56 | 96.2 | Field |
| 34.25 | 31.00 | 1.10 | 93.5 | " |
| 17.12 | 15.50 | 1.10 | 86.6 | " |
| 6.80 | 22.00 | 0.37 | 47.8 | " |
| 50.80 | 7.10 | 7.15 | 71.5 | Davison |

This table shows the influence of metal content and metal ratio on the composition of the deposit. It is seen that the copper

varies from 3.17 to 50.80 g. per liter; the zinc from 6.53 to 31.00 g. per liter; and the total metal content from 12.88 to 65.25 g. per liter. The slight influence which the metal content has on the composition of the deposit is shown by the fact that Spitzer obtained a deposit of 70 percent copper from a solution which contained 3.17 g. of copper, while Davison obtained a deposit of only 71.5 percent copper from a solution which contained 50.80 g. of copper. Field found that by diluting a solution which gave a deposit of 93.5 percent copper with an equal volume of water, the percentage of copper in the deposit was reduced to 86.6. From this it is evident that factors other than the absolute metal content and the ratio of copper to zinc in the solution influence the composition of the deposit to a marked degree.

A similar analysis of the results obtained by them on the influence of cyanide content, alkalinity, current density, and temperature makes clearly evident the fact that our present knowledge of the brass plating solution is decidedly indefinite and incomplete. The lack of specific data regarding the influence of each of these factors, together with the need for such data, owing to the extensive use of the brass plating solution commercially, makes it appear that a thorough investigation of the influence of each of these factors is highly desirable.

Each factor in some way affects the metal ion concentration of the solution. All the effects produced by changes in metal ratio, metal content, cyanide content, alkalinity, current density, and temperature must ultimately result from changes in metal ion concentration. Since the influence of all of these factors is the result of a change in metal ion concentration, it seems that a study of metal ion concentrations is the most logical method of attacking this problem. Such a study is most successfully made by potential measurements. For this reason, therefore, the influence of each of these factors upon the single potentials of copper and zinc in their cyanide solutions was determined.

## *Theoretical.*

A difference of potential exists between an ionizable substance and a solution of its ions. The magnitude of this potential in the case of a metal is given by the well-known equation

$$E = \frac{RT}{NQ} \log_e \frac{P}{p}. \qquad (1)$$

In this equation $E$ is the potential in volts which a metal of solution tension $P$ and valence $N$ shows against a solution of one of its salts in which the osmotic pressure of the metal ions is represented by $p$. The absolute temperature is represented by $T$, Faraday's constant by $Q$, and the gas constant by $R$. $E$ is the equilibrium potential which must be exceeded before metal ions will deposit from such a solution. Metals which become positively charged in a solution, normal with respect to their ions, will be known as electro-positive in this discussion. Those which assume a negative charge will be known as electro-negative.

In the electro-deposition of a metal from an aqueous solution of its ions it is necessary to keep in mind that hydrogen ions are also present. If the applied potential exceeds a certain value hydrogen will be deposited. The magnitude of this potential is given by the equation

$$E_1 = e_h + \frac{RT}{Q} \log_e \frac{P'}{p'} \qquad (2)$$

where $e_h$ is the hydrogen over-voltage for the particular electrode, $P'$ is the solution potential of hydrogen and $p'$ is the osmotic pressure of hydrogen ions in the solution. The other letters in the equation have the same significance as in equation (1). According to equation (1) the potential necessary to separate the metal is

$$E = \frac{RT}{NQ} \log_e \frac{P}{p}.$$

It is evident that if $E_1$ is greater than $E$ metal only will be deposited, if $E_1$ is equal to $E$ hydrogen and the metal will separate simultaneously, and if $E_1$ is less than $E$ hydrogen alone will be deposited. This reasoning applies equally well for any other positive ion in the solution.

From this it follows that, from a solution in which there are a number of metallic ions, only that metal is deposited which is the most electro-positive. For example, in an ordinary solution containing copper and zinc ions, copper alone is deposited, since copper is nearly a volt more positive than zinc.

In order to separate two metals simultaneously, as pointed out above, the single potentials which these metals show in the solution must be nearly equal. Since the solution potential of a metal has a definite characteristic value, it follows from equation (1) that the potential which a metal shows in a particular solution must depend upon the osmotic pressure of its ions in that solution. If in the above solution of copper and zinc sulphate, the concentration of the copper ions be made continuously smaller, the copper becomes less electro-positive and thus approaches the value of zinc in the same solution. If this process could be continued long enough zinc and copper would deposit simultaneously. The practical impossibility of accomplishing this by dilution is indicated by a calculation from equation (1) which shows that the copper ion concentration in such a solution is only about $1 \times 10^{-38}$ N.

The decrease in metal ion concentration, by dilution, decreases also the conductivity. Both the low metal content and the low conductivity thus produced are undesirable. When metal content is low there is no ready source for the renewal of the ions removed from about the cathode, and they are practically all removed by the passage of only a small quantity of electricity. When the conductivity is low a greater amount of energy is consumed in the deposition of a given amount of metal. In order, therefore, to decrease the ion concentration of a solution without decidedly decreasing the conductivity, and at the same time to provide a ready source of ion supply, substances are used which form complex ions containing the metal. For example, the addition of sodium cyanide to a solution of copper sulphate forms the complex salt $Na_2Cu(CN)_3$, which dissociates until the following equilibrium is established—

$$Na_2Cu(CN)_3 \leftrightharpoons 2Na^+ + \overset{=}{Cu}(CN)_3 \leftrightharpoons 2Na^+ + Cu^+ + 3\overset{-}{C}N.$$

Equilibrium is established with an extremely small concentration of copper ions. Zinc forms a similar complex salt $Na_2Zn(CN)_4$ and a similar equilibrium is established—

$$Na_2Zn(CN)_4 \leftrightharpoons 2Na^+ + \overset{=}{Zn}(CN)_4 \leftrightharpoons 2Na^+ + Zn^{++} + 4\overset{-}{C}N.$$

In case copper or zinc are removed from such solutions, new ions, formed by dissociation, immediately replace them. The

copper complex compound is much more stable than the zinc, hence, by the addition of sodium cyanide to a solution of copper and zinc sulphates, the copper ion concentration is decreased decidedly more than the zinc (Spitzer, *loc. cit.*). This means that the single potentials of zinc and copper approach each other on the addition of sodium cyanide. By the addition of large quantities of sodium cyanide the potentials become equal, or copper may even become more electro-negative than zinc. (Spitzer, *loc. cit.* S. P. Thompson, Chem. News (1887), **55**, 300.) The copper ion concentration in such a solution is about $10^{-38}$ N.

From this it appears possible to prepare a solution of the complex cyanide compounds of copper and zinc in which these metals show the same single potentials. During the process of electrolysis of such a solution the single potentials of copper and zinc are different from the equilibrium value. This results from the change in ion concentration immediately surrounding the electrodes. The actual ion concentration in the vicinity of the electrodes depends upon the current density, temperature, rate of stirring, and rate of ionization of the complex compounds. Other conditions remaining the same, it is evident that an increase in current density results in an increase in the metal ion concentration at the anode and a decrease at the cathode. Increase in temperature, by increasing diffusion, counteracts to some extent these changes in ion concentration. Stirring and ionization also tend to maintain a uniform ion concentration.

The value of electrolytically deposited brass is determined chiefly by its color and this depends largely upon its composition. A brass of 65 percent copper and 35 percent zinc has, under favorable conditions, a satisfactory, bright yellow appearance. In order that the composition of the solution which deposits brass of this composition may remain unaltered the metals must dissolve from the anode in this ratio and the efficiency of solution must be the same as that of deposition. These are the conditions which must be fulfilled by an efficient brass plating solution and the maintenance of these conditions constitutes the chief problem of brass plating. The ratio in which copper and zinc dissolve or deposit is determined by the ratio of their ion concentrations in the solution immediately surrounding the electrode. The problem, then, consists in establishing this ratio and in maintaining

it by the proper regulation of factors which influence ion concentration.

*Materials and Method of Manipulation.*

The cells used were 600 c.c. beakers. There were two anodes and one cathode in each cell. One anode was a sheet of cast zinc 50 by 50 by 2 mm. and the other was a sheet of electrolytic copper of the same dimensions. These were supported parallel to each other and about 8 cm. apart. The cathode was a platinum sheet 50 mm. square, supported midway between the anodes. Between each anode and the positive terminal of the battery were placed a coulometer, a variable resistance, and an ammeter. A return wire from the cathode to the negative terminal of the battery completed the circuit.

The solution was vigorously stirred by a glass rod provided with a paddle about 3 cm. long. The rod was rotated about 1200 r. p. m.

The potential measurements were made by the compensation method with a small Leeds and Northrup potentiometer graduated to 0.5 millivolt. A suitable galvanometer was used as zero instrument. As a standard comparison potential, a cadmium cell with a potential of 1.01845 at 20° C. was used. Normal calomel electrodes were used as reference electrodes. To avoid including in the potential measurements some of the potential fall due to the current in the solution, the ends of the calomel electrodes were placed as near the anodes as possible and on the side opposite to the cathode. The end of the calomel electrode against which the cathode potential was measured was placed near the edge of the cathode for the same reason.

Only the highest grade C. P. materials were used in this work. Analysis showed that they did not require further purification.

Two cells were set up as above described, and all determinations were run in duplicate. Only one set of results for each determination, however, is recorded in the tables. At the beginning of each determination the anodes and cathodes were weighed and placed in the cells. The calomel electrodes were placed in position and a set of potential readings taken before the current was turned on. The current density was 0.15 ampere per sq. dm. at each anode and 0.30 ampere at the cathode. The current was

allowed to flow for two hours, giving a cathode deposit of about 0.6 g. With the current flowing, three sets of potential readings were taken, one immediately after the current was turned on, one at the middle of the determination, and one just before the current was turned off. Immediately after the current was turned off another set of potential readings was taken. All these readings are recorded in the tables.

At the end of the determination the anodes and cathode were immediately removed from the solution, washed with tap water, with distilled water, and finally with alcohol. They were then allowed to dry at room temperature before weighing. From the loss in weight of the anodes and the quantity of electricity as shown by the coulometers, the efficiencies of anode corrosion were calculated.

The deposit was dissolved in dilute nitric acid and the copper determined electrolytically. Zinc in the deposit was obtained by difference. From these data the percentage of copper in the deposit and the cathode efficiency were calculated. The efficiency of corrosion or deposition is obtained by dividing the weight of material dissolved or deposited by the weight of material equivalent to the amount of electricity shown by the coulometers.

Unless otherwise stated, the temperature was 25° C. ± 1°. A closer regulation of the temperature was not considered necessary.

*Metal Ratio.*

It has already been pointed out that there are many factors which influence the metal ion concentration of the brass plating solution and the nature of the deposit. These factors were individually studied and the exact influence of each determined. Metal ratio was the first of these factors to receive special attention.

It seemed logical to begin with a solution containing the two metals in about the same ratio. The solution first used contained 4.5 grams of copper and 6.7 grams of zinc per liter in the form of complex cyanides. (This is the metal content of solution 4 in Bennett's (*loc. cit.*) table.) No other constituents were present. The solution was electrolyzed for twenty hours and the changes which resulted in it observed. These observations are recorded in Table II.

The many disadvantages of this solution are readily apparent. The results show at first a high anode solution due to the corrosive action of the excess cyanide. The excess cyanide also produces a high cathode polarization which causes the deposition of large quantities of hydrogen and consequently a low cathode efficiency. As electrolysis is continued the excess cyanide is removed; this, as is to be expected, increases the cathode efficiency and decreases the zinc anode efficiency. The potentials of both copper and zinc decrease. In cyanide solutions the solution pressure of these metals is greater than the osmotic pressure of their ions, hence a decrease in the potentials indicates an increase in

TABLE II.

| Row | Efficiencies | | | Per-cent of Copper in Deposit | Grams of Free Cyanide per Liter | Total Hrs. Run | Potential Measurements | | | | Character of Deposit |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | Anodes | | Cath. | | | | With Current | | Without Current | | |
| | Copper | Zinc | | | | | Copper | Zinc | Copper | Zinc | |
| 1 | 108.0 | 87.2 | 6.9 | 45.5 | 7.4 | 2 | 0.951 | 1.231 | 1.073 | 1.272 | Dull Red |
| 2 | 107.2 | 100.4 | 75.8 | 58.9 | 5.4 | 4 | 0.906 | 1.115 | 1.059 | 1.248 | " " |
| 3 | 107.2 | 129.3 | 91.2 | 57.4 | 4.5 | 6 | 0.822 | 1.100 | 0.987 | 1.241 | Uniform Yellow |
| 4 | 109.3 | 96.7 | 96.9 | 39.9 | 3.6 | 8 | 0.834 | 1.129 | 0.992 | 1.228 | " " |
| 5 | 114.4 | 94.1 | 100.2 | 36.5 | 3.3 | 10 | 0.814 | 1.149 | 0.983 | 1.231 | Purple |
| 6 | 107.5 | 116.2 | 98.7 | 43.7 | 3.5 | 12 | 0.679 | -0.531 | 0.826 | 0.891 | Dark Brown |
| 7 | 107.6 | 57.1 | 95.3 | 48.3 | 3.5 | 14 | 0.660 | -0.595 | 0.864 | 0.940 | " " |
| 8 | 106.5 | 49.2 | 96.4 | 44.7 | 3.0 | 16 | 0.655 | -0.575 | 0.870 | 0.795 | " " |
| 9 | 108.8 | 55.8 | 97.0 | 45.8 | 2.9 | 18 | 0.654 | -0.573 | 0.817 | 0.705 | " " |
| 10 | 108.8 | 60.3 | 97.1 | 47.7 | 2.6 | 20 | 0.606 | -0.676 | 0.804 | 0.665 | " " |

metal ion concentration. The change in ion concentration is more pronounced in the case of zinc than of copper; so much more, in fact, that the copper becomes more electro-negative than zinc. The change appears to be most pronounced at a definite stage in the electrolysis. At this stage a white precipitate of zinc cyanide appears on the zinc anode and the efficiency of this anode suddenly decreases. This change in relative ion concentration is probably responsible for the fluctuation in the percentage of copper in the deposit.

The percentage of copper in all the deposits obtained here was lower than that required for a good brass. This low percentage of copper indicated that the copper content of the solution was too

low. It seemed, therefore, that by adding copper, a solution might be obtained which would give a deposit of suitable copper content in the presence of sufficient free cyanide to produce efficient anode corrosion. The addition of copper, as the complex cyanide, to the electrolyzed solution, showed that the percentage of copper in the deposit could be increased in this way. The results of such additions are given in Table III.

A solution of the composition of that used in Table II was electrolyzed nearly to the stage at which the decided change in ion concentration occurred. It then gave the values in row 1 of Table III. To this solution copper sulphate, with the equivalent sodium cyanide, was added in sufficient quantity to double the original

TABLE III.

| Row | Efficiencies | | | Per-cent of Copper in Deposit | Grams of Copper per Liter of Sol. | Potential Measurements | | | | Character of Deposit |
|---|---|---|---|---|---|---|---|---|---|---|
| | Anodes | | Cath. | | | With Current | | Without Current | | |
| | Copper | Zinc | | | | Copper | Zinc | Copper | Zinc | |
| 1 | 103.8 | 95.5 | 96.7 | 36.7 | 4.46 | 0.640 | 1.015 | 0.865 | 1.189 | Dull yellow |
| 2 | 104.7 | 106.5 | 80.1 | 59.1 | 8.92 | 0.750 | 0.967 | 0.964 | 1.130 | " " |
| 3 | 108.1 | 102.8 | 76.5 | 62.4 | 13.37 | 0.861 | 0.918 | 0.926 | 1.125 | Bright " |
| 4 | 109.1 | 100.3 | 99.0 | 66.1 | 17.83 | 0.869 | 0.994 | 0.934 | 1.009 | Dull " |
| 5 | 109.7 | 104.7 | 98.7 | 80.7 | 23.40 | 0.854 | 0.991 | 0.902 | 1.027 | Bright " |

copper content. The effect this had on the solution is indicated in row 2. Further additions of copper were made and the results obtained are given in rows three, four and five. The increase in copper concentration is accompanied by the following changes: (1) the percentage of copper in the deposit increases, and as might be expected the nature of the deposit becomes more satisfactory; (2) the potential of zinc decreases, indicating an increase in zinc ion concentration; (3) the potential of copper increases, indicating a decrease in the copper ion concentration. This decrease in copper ion concentration is not exceptional, since in all electrolytes the ion concentration becomes less with increase in concentration of the electrolyte beyond a certain point. This phenomenon in the case of copper has also been observed by Spitzer (*loc. cit.*). Copper tends to form complex compounds

containing more cyanide than $Na_2Cu(CN)_3$; and since the copper complex is more stable than the zinc, the latter will tend to be decomposed in a solution which contains both. This will allow more zinc cyanide to dissociate and hence increase the zinc ion concentration.

From the table it appears that, with a solution which contained 23.4 g. of copper and 6.7 g. of zinc, a deposit of the desired copper content can be obtained with approximately 100 percent anode and cathode efficiencies. Accordingly, a solution which contained 24.0 g. of copper and 6.7 g. of zinc was prepared, but it was found that this solution, when boiled for a half hour, gave a deposit of only 60.0 percent copper. All solutions prepared at

TABLE IV.

| Row | Efficiencies | | | Per-cent of Copper in De-posit | Grams of Cop-per per Liter of Sol. | Potential Measurements | | | | | | Character of Deposit |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Anodes | | Cath. | | | With Current | | | Without Current | | | |
| | | | | | | Anodes | | Cath. | Anodes | | Cath. | |
| | Copper | Zinc | | | | Copper | Zinc | | Copper | Zinc | | |
| 1 | 105.5 | 117.4 | 97.6 | 60.1 | 24.46 | 0.900 | 0.878 | 1.444 | 0.974 | 1.076 | 0.973 | Dull Yellow |
| 2 | 107.2 | 104.4 | 96.4 | 67.7 | 28.46 | 0.874 | 0.942 | 1.463 | 0.963 | 1.085 | 0.987 | " " |
| 3 | 104.0 | 90.8 | 100.0 | 75.2 | 32.46 | 0.892 | 0.922 | 1.388 | 0.940 | 1.030 | 0.960 | " Red |
| 4 | 102.8 | 96.3 | 98.8 | 80.9 | 37.00 | 0.875 | 0.942 | 1.319 | 0.936 | 1.037 | 0.987 | " " |

room temperature had a characteristic red-brown color which was removed by boiling and also by continued electrolysis. From these results it appeared that there was a close connection between the removal of the red-brown color and the decrease in the percentage of copper in the deposit, and that the changes within the solution, which are responsible for the decrease in the percentage of copper in the deposit, may be brought about by boiling the solution as well as by continued electrolysis. The potentials obtained in the boiled solution corresponded to those obtained in an unboiled solution of lower copper content. This is in agreement with the results of Hönig (*loc. cit.*) who found that the separation potential of copper from its cyanide solution was increased to a marked extent by boiling the solution. The exact nature of the change produced is unknown.

These results indicated that in order to obtain the desired percentage of copper in the deposit still greater concentrations of copper were necessary. Four solutions, containing respectively 24.5, 28.5, 32.5 and 37.0 g. of copper, were prepared and boiled

TABLE V.

| Row | Efficiencies | | | Percent of Copper in Deposit | Number of Hours Electrolyzed | Potential Measurements | | | | | | Character of Deposit |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Anodes | | Cath. | | | With Current | | | Without Current | | | |
| | | | | | | Anodes | | Cath. | Anodes | | Cath. | |
| | Copper | Zinc | | | | Copper | Zinc | | Copper | Zinc | | |
| 1 | 107.0 | 85.2 | 93.6 | 64.0 | 2 | 0.891<br>0.897<br>0.903 | 0.976<br>0.960<br>0.980 | 1.420 | 0.955<br>0.979 | 1.064<br>1.080 | 1.000 | Dull light yellow |
| 2 | 105.6 | 87.9 | 98.4 | 64.9 | 6 | 0.883<br>0.883<br>0.884 | 0.950<br>0.950<br>0.948 | 1.381 | 0.956<br>0.967 | 1.039<br>1.051 | 0.996 | Dull yellow |
| 3 | 105.6 | 103.3 | 98.5 | 65.9 | 10 | 0.858<br>0.864<br>0.867 | 0.980<br>0.983<br>0.986 | 1.370 | 0.941<br>0.958 | 1.045<br>1.032 | 0.987 | " " |
| 4 | 103.9 | 92.2 | 99.0 | 68.4 | 18 | 0.873<br>0.869<br>0.867 | 0.956<br>0.939<br>0.948 | 1.365 | 0.947<br>0.955 | 1.030<br>1.054 | 0.981 | " " |
| 5 | 104.1 | 93.4 | 98.3 | 68.2 | 34 | 0.875<br>0.875<br>0.879 | 0.954<br>0.983<br>0.978 | 1.346 | 0.951<br>0.957 | 1.075<br>1.108 | 0.983 | " " |
| 6 | 104.4 | 96.2 | 98.1 | 63.3 | 54 | 0.887<br>0.890<br>0.887 | 1.035<br>0.985<br>0.959 | 1.368 | 0.963<br>0.973 | 1.085<br>1.119 | 0.997 | " " |
| 7 | 105.0 | 107.9 | 103.3 | 50.8 | 72 | 0.853<br>0.841<br>0.837 | 0.900<br>0.869<br>0.891 | 1.371 | 0.935<br>0.950 | 1.090<br>1.107 | 1.023 | Bright yellow |
| 8 | 104.1 | 106.0 | 104.2 | 50.3 | 74 | 0.847<br>0.836<br>0.829 | 0.805<br>0.807<br>0.803 | 1.358 | 0.933<br>0.931 | 1.100<br>1.107 | 0.994 | " " |
| 9 | 103.2 | 108.8 | 103.5 | 42.1 | 88 | 0.755<br>0.763<br>0.770 | 0.377<br>0.384<br>0.389 | 1.394 | 0.860<br>0.891 | 1.201<br>1.215 | 1.195 | " silver color |

previous to electrolysis. All these solutions contained 6.7 g. of zinc per liter. These solutions on electrolysis gave the results recorded in Table IV. The data here given show that an increase in the ratio of copper to zinc in the solution increases the percentage of copper in the deposit. A solution containing 28.5 g. of copper per liter is necessary under these conditions to give a deposit containing 65 percent of copper.

To determine whether or not a solution of this composition was suitable for use in the remainder of this investigation, two samples were electrolyzed for a period of eighty-eight hours. At various stages in the electrolysis determinations were made of the cathode and anode efficiencies and of the percentage of copper in the deposit. The usual potential readings were taken. The results are recorded in Table V.

These results show that a solution of this composition gives satisfactory anode and cathode efficiencies for a period of about fifty hours. The high copper anode efficiencies may be accounted for by the solvent action of the cyanide on copper. The zinc anode efficiencies recorded are not as reliable as those for copper, because in nearly all cases copper deposited on the zinc anode and decreased the accuracy of the results. The constancy of the ion concentrations during this period is indicated by the constancy of the potential measurements.

Satisfactory bright yellow brass deposits are obtained during the first fifty hours. Later the deposits become slightly dull and at the end of eighty-eight hours they have a pink appearance. It is noticed that even in this solution the percentage of copper in the deposit finally decreases. Since, however, this solution can be relied upon to give constant results for a period of about fifty hours, it is satisfactory for use in the remainder of this work.

### *Metal Content.*

The influence of a decrease in metal content of the electrolyte was next determined. Two dilutions were made; one by adding an equal volume of water to the original solution, and the other by adding an equal volume of water to the diluted solution. The usual potential measurements and efficiency determinations were made and the data recorded in Table VI.

The potential measurements show that as the solution is diluted there is practically no change in zinc ion concentration, but an increase in copper ion concentration. The increase in the degree of ionization of the copper salt on dilution must increase the copper ion concentration more than dilution decreases it. The increase in copper ion concentration would normally increase the percentage of copper in the deposit, but the increase in cathode polarization shows that the metal ions are removed by electro-

deposition more rapidly than they are furnished by dissociation in these dilute solutions. This results in the deposition of relatively greater amounts of the more electro-negative metal, zinc. In the second dilution, however, this effect is overcome by the more rapid increase in copper ion concentration. The increase in cathode polarization is accompanied by the usual decrease in cathode efficiency.

During the electrolysis of the dilute solutions white deposits of metal cyanides form on the anodes. This indicates that the

TABLE VI.

| Row | Efficiencies | | | Per-cent of Copper in Deposit | Con-centra-tion, Origi-nal | Potential Measurements | | | | | | Character of Deposit |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Anodes | | Cath. | | | With Current | | | Without Current | | | |
| | | | | | | Anodes | | Cath. | Anodes | | Cath. | |
| | Copper | Zinc | | | | Copper | Zinc | | Copper | Zinc | | |
| 1 | 103.2 | 103.9 | 97.1 | 66.6 | ..... | 0.872<br>0.867<br>0.869 | 1.007<br>1.000<br>1.006 | 1.485<br>1.495<br>1.500 | 0.946<br>0.976 | 1.046<br>1.052 | 1.022 | Dull red |
| 2 | 108.0 | 108.3 | 92.2 | 59.5 | ½ | 0.844<br>0.831<br>0.830 | 0.983<br>0.963<br>0.930 | 1.555<br>1.564<br>1.550 | 0.937<br>0.950 | 1.135<br>1.080 | 0.996 | " gray |
| 3 | 68.2 | 73.8 | 81.3 | 61.0 | ¼ | 0.795<br>—0.535<br>—0.550 | 0.915<br>—0.760<br>—0.690 | 1.619<br>1.625<br>1.621 | 0.900<br>0.921 | 1.100<br>1.140 | 0.980 | " " |

cyanide concentration is too low, and accounts for the low anode efficiencies. The results obtained here are due not only to the decrease in metal content but also to the decrease in the cyanide concentration. It is clearly evident from these results that the original solution is the most satisfactory.

*Current Density.*

The original solution, when electrolyzed at different current densities, gave the results recorded in Table VII.

The data show that both the cathode efficiency (Curve 1, Plate I) and the percentage of copper in the deposit (Curve 3, Plate I) uniformly decrease and that the cathode polarization (Curve 2,

Plate I) uniformly increases with increase in current density. The potential measurements show that the ion concentrations remain practically unchanged. The decrease in cathode efficiency and percentage of copper in the deposit are to be expected, since

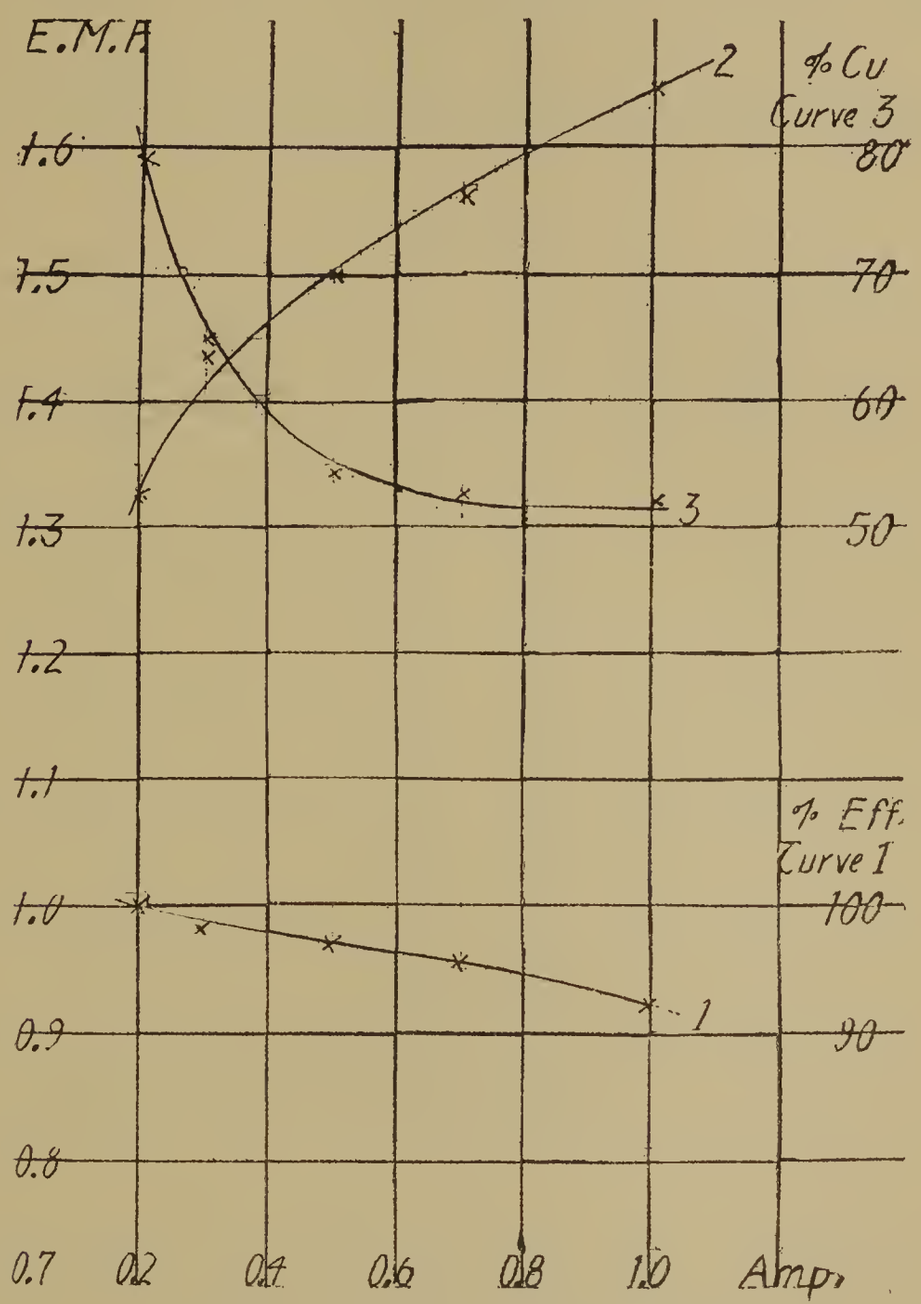


PLATE I. Influence of Current Density.
1. Change in cathode efficiency with increase in current density.
2. Potential of copper anode with current flowing.
3. Potential of cathode with current flowing.

an increased cathode polarization produces the deposition of relatively larger quantities of zinc and hydrogen. Neither a maximum percentage of copper nor a minimum cathode efficiency, such as was obtained by Spitzer (*loc. cit.*), is here observed. This is probably due to the difference between the metal content of this solution and that used by Spitzer.

The character of the deposit becomes less satisfactory with increase in current density beyond 0.3 ampere per sq. dm. of cathode surface. For this reason a current density of 0.3 ampere is used in the remainder of this work.

*Temperature.*

The work of other investigators shows that an increase in temperature increases the percentage of copper in the deposit and the

TABLE VII.

| Row | Efficiencies | | | Per-cent of Copper in Deposit | Current Density Amp./dm.² | Potential Measurements | | | | | | Character of Deposit |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Anodes | | Cath. | | | With Current | | | Without Current | | | |
| | | | | | | Anodes | | Cath. | Anodes | | Cath. | |
| | Copper | Zinc | | | | Copper | Zinc | | Copper | Zinc | | |
| 1 | 104.6 | 91.4 | 100.5 | 78.6 | 0.2 | 0.907<br>0.893<br>0.894 | 0.950<br>0.943<br>0.937 | 1.328 | 0.978<br>0.966 | 1.070<br>1.042 | 0.991 | Dull red |
| 2 | 102.9 | 89.2 | 98.2 | 64.8 | 0.3 | 0.852<br>0.853<br>0.849 | 0.941<br>0.937<br>0.941 | 1.440 | 0.966<br>0.959 | 1.091<br>1.064 | 0.989 | Dull red, not uniform |
| 3 | 105.8 | 97.7 | 97.0 | 53.8 | 0.5 | 0.862<br>0.858<br>0.859 | 0.968<br>0.949<br>0.938 | 1.500 | 0.983<br>0.985 | 1.130<br>1.082 | 1.009 | Dull gray |
| 4 | 102.0 | 112.2 | 95.7 | 52.9 | 0.7 | 0.858<br>0.858<br>0.864 | 0.942<br>0.954<br>0.961 | 1.560 | 0.984<br>0.985 | 1.100<br>1.056 | 1.004 | " " |
| 5 | 100.9 | 101.6 | 92.4 | 52.1 | 1.0 | 0.795<br>0.791<br>0.787 | 0.540<br>—0.744<br>—0.880 | 1.651 | 0.986<br>0.979 | 1.097<br>1.093 | 0.999 | " " |

cathode efficiency. Since the compositions of the solutions used by them were much different from the one here used, and since no potential measurements were made, it was considered desirable to obtain a series of measurements at different temperatures. The results are given in Table VIII.

It is observed that with increase in temperature there is a slight gradual increase in the potentials of copper and zinc in the solution (Curves 1 and 2, Plate II). This is probably accounted for by the more rapid diffusion of the dissolved material away from

the electrodes at higher temperatures. The pronounced decrease in the cathode polarization with increase in temperature (Curve 3, Plate II) probably results from the rapid diffusion of metal ions to the cathode. Accompanying the decrease in cathode polarization is the usual increase in the percentage of copper in the deposit (Curve 4, Plate II).

These results are in good agreement with those obtained by Field (*loc. cit.*) and others.

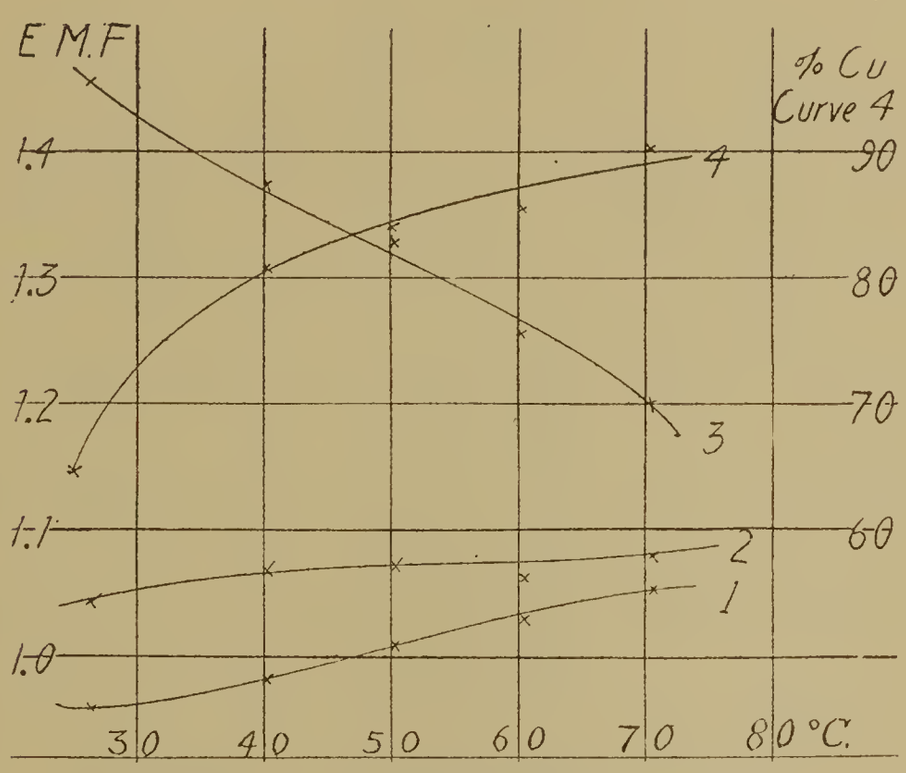


PLATE II. Influence of change in Temperature.
1. Potential of copper without current.
2. Potential of zinc without current.
3. Potential of cathode with current flowing.
4. Change in the percentage of copper in the deposit.

## *Sodium Cyanide.*

The results obtained when sodium cyanide was added to the solution are given in Table IX.

A study of the literature gives the impression that an increase in free cyanide increases the anode efficiency. These results, however, fail to show this effect. In fact, the efficiency of the zinc anode tends to decrease with increase in free cyanide. As the concentration of the free cyanide increases, the potentials of the anodes and the cathode increase (Curves 1, 2 and 3, Plate III); this indicates a decrease in metal ion concentration. This

decrease in metal ion concentration makes necessary an increase in cathode potential in order to deposit the metals. The increase in cathode potential causes the deposition of relatively larger quantities of hydrogen and results in a decrease in cathode efficiency.

The percentage of copper in the deposit shows a peculiar change. There is first a decided decrease, then a gradual increase as the concentration of free cyanide increases (Curve 4, Plate III).

TABLE VIII.

| Row | Efficiencies | | | Per-cent of Copper in Deposit | Tem-pera-ture Cent. | Potential Measurements | | | | | | Character of Deposit |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Anodes | | Cath. | | | With Current | | | Without Current | | | |
| | | | | | | Anodes | | Cath. | Anodes | | Cath. | |
| | Copper | Zinc | | | | Copper | Zinc | | Copper | Zinc | | |
| 1 | 103.1 | 88.0 | 98.1 | 64.2 | 26° | 0.865<br>0.868<br>0.868 | 0.943<br>0.960<br>0.933 | 1.456 | 0.960<br>0.957 | 1.054<br>1.043 | 0.992 | Dull red |
| 2 | 105.4 | 93.2 | 100.2 | 80.9 | 40° | 0.927<br>0.928<br>0.936 | 0.983<br>0.960<br>0.962 | 1.371 | 0.976<br>0.982 | 1.083<br>1.053 | 1.023 | Bright red-yellow |
| 3 | 104.8 | 72.0 | 100.7 | 83.9 | 50° | 0.958<br>0.965<br>0.972 | 0.991<br>0.997<br>1.007 | 1.327 | 1.000<br>1.007 | 1.070<br>1.056 | 1.037 | Bright red-yellow |
| 4 | 104.6 | 83.7 | 99.2 | 85.2 | 60° | 1.008<br>1.006<br>1.009 | .984<br>1.007<br>1.009 | 1.253 | 1.026<br>1.031 | 1.056<br>1.056 | 1.040 | Bright red-yellow |
| 5 | 103.8 | 57.7 | 99.6 | 90.2 | 70° | 1.041<br>1.039<br>1.038 | 1.045<br>1.044<br>1.040 | 1.200 | 1.046<br>1.055 | 1.078<br>1.078 | 1.062 | Bright red-yellow |

This could not be accounted for by observed changes in potential and was at first thought to be due to error in analysis. Two other sets of determinations, the data for which are not given, showed a similar behavior. It thus appears that there are two concentrations of free cyanide at which a brass containing 65 percent of copper (a satisfactory composition) can be obtained from this solution. Since the lower cyanide concentration gives a much better cathode efficiency and an equally good anode efficiency, it is the one used in the remainder of this work.

*Acid Substances.*

The greater part of the copper and zinc in their cyanide solutions is present as $Na_2Cu(CN)_3$ and $Na_2Zn(CN)_4$ (F. Kunchert, Zeitsch. anorg. Chem. (1904), **41**, 337). A study of the various possible equilibria that may exist in such solutions shows that acid and alkaline substances may influence the extent to which

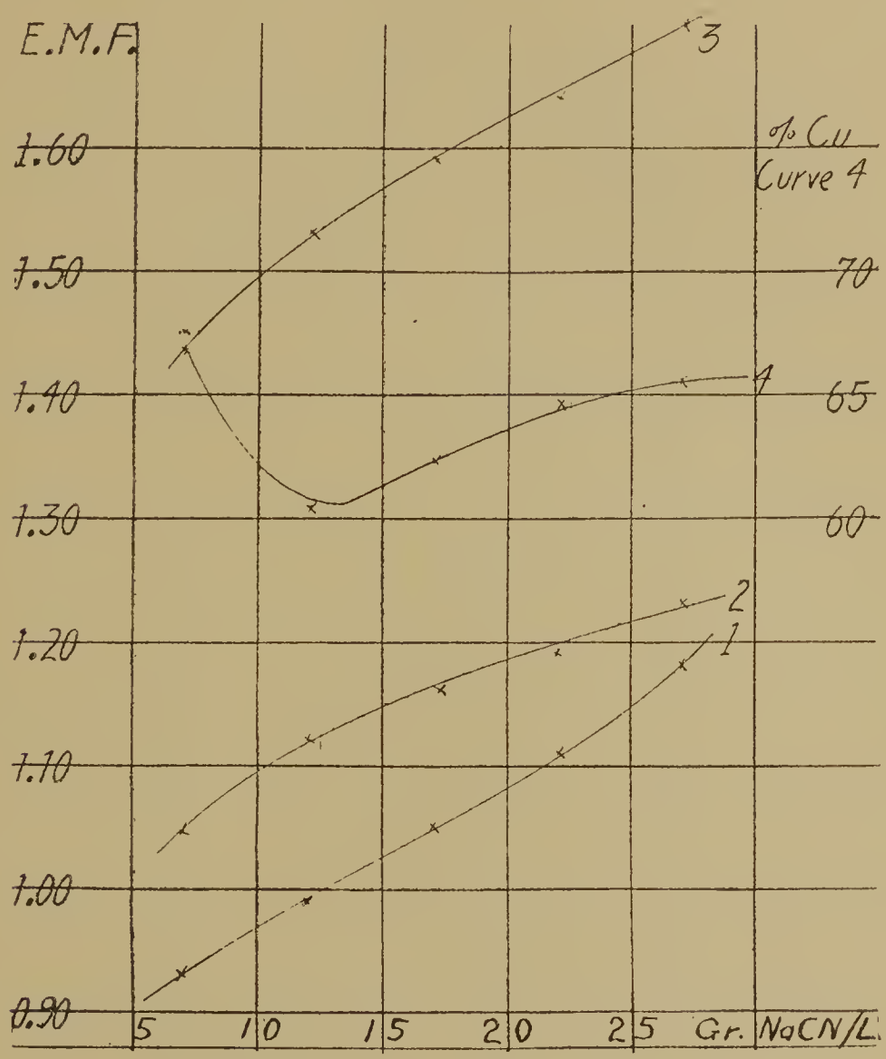


PLATE III. Influence of Sodium Cyanide.

1. Potential of copper anode without current.
2. Potential of zinc anode without current.
3. Potential of cathode with current.
4. Percentage of copper in the deposit.

the indicated reactions take place and hence the metal ion concentration in these solutions. (See next page.)

It is apparent that the addition of alkaline substances forces reactions (I *a 2*) and (II *a 2*) to the left; this increases the concentration of molecular sodium cyanide. Reactions (I *a 1*) and (II *a 1*) are then forced to the right, increasing the concentration

$$
\begin{array}{lll}
 & & 2Na^{+} + 2\bar{C}N \qquad (1) \\
 & & \Downarrow\Uparrow \\
 & (a)\ \left(CuCN \leftrightharpoons Cu^{+} + \bar{C}N\right) + (2NaCN + 2H_2O & \\
 & \Downarrow\Uparrow & \Downarrow\Uparrow \\
\text{I.}\ 2H_2O + Na_2Cu(CN)_3 & & 2NaOH + 2HCN) \qquad (2) \\
 & \Downarrow\Uparrow & \\
 & (b)\ 2Na^{+} + \left(\bar{\bar{C}}u(CN)_3 \leftrightharpoons Cu^{+} + 3\bar{C}N\right) &
\end{array}
$$

$$
\begin{array}{lll}
 & & 2Na^{+} + 2\bar{C}N \qquad (1) \\
 & & \Downarrow\Uparrow \\
 & (a)\ \left(Zn(CN)_2 \leftrightharpoons Zn^{++} + 2\bar{C}N\right) + 2NaCN + 2H_2O & \\
 & \Downarrow\Uparrow & \Downarrow\Uparrow \\
\text{II.}\ 2H_2O + Na_2Zn(CN)_4 & & 2NaOH + 2HCN) \qquad (2) \\
 & \Downarrow\Uparrow & \\
 & (b)\ 2Na^{+} + \left(\bar{\bar{Z}}n(CN)_4 \leftrightharpoons Zn^{++} + 4\bar{C}N\right) &
\end{array}
$$

of sodium and cyanide ions. This increase in sodium and cyanide ions from the sodium cyanide causes a decrease in the concentration of copper and zinc ions.

The addition of acid substances forces reactions (I *a 2*) and (II *a 2*) to the right with the formation of slightly dissociated hydrocyanic acid, thus increasing the cyanide ion concentration

TABLE IX.

| Row | Efficiencies | | | Per-cent of Copper in Deposit | Grams of Free Cyanide per Liter | Potential Measurements | | | | | | Character of Deposit |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Anodes | | Cath. | | | With Current | | | Without Current | | | |
| | | | | | | Anodes | | Cath. | Anodes | | Cath. | |
| | Copper | Zinc | | | | Copper | Zinc | | Copper | Zinc | | |
| 1 | 103.2 | 103.9 | 97.1 | 66.6 | 7.0 | 0.850<br>0.870<br>0.880 | 0.956<br>0.965<br>0.981 | 1.439<br>1.435<br>1.436 | 0.921<br>0.955 | 1.052<br>1.040 | 0.989 | Dull red |
| 2 | 103.6 | 87.5 | 94.7 | 60.4 | 12.0 | 0.879<br>0.882<br>0.883 | 1.024<br>1.009<br>1.006 | 1.539<br>1.542<br>1.546 | 0.990<br>1.002 | 1.125<br>1.055 | 1.035 | " gray |
| 3 | 103.7 | 74.1 | 79.6 | 62.3 | 17.0 | 0.943<br>0.926<br>0.929 | 1.060<br>1.067<br>1.060 | 1.595<br>1.590<br>1.595 | 1.057<br>1.062 | 1.169<br>1.142 | 1.095 | " " |
| 4 | 102.7 | 86.3 | 51.3 | 64.8 | 22.0 | 0.971<br>0.960<br>0.950 | 1.111<br>1.105<br>1.104 | 1.650<br>1.642<br>1.644 | 1.126<br>1.111 | 1.189<br>1.183 | 1.146 | " " |
| 5 | 103.3 | 86.5 | 38.2 | 65.5 | 27.0 | 1.065<br>1.051<br>1.013 | 1.195<br>1.166<br>1.130 | 1.696<br>1.707<br>1.712 | 1.200<br>1.164 | 1.245<br>1.220 | 1.174 | " " |

produced from the sodium cyanide. This permits a greater dissociation of the copper and zinc complexes and results in an increase in the concentration of copper and zinc ions. It should be mentioned here that, since the stability of the copper complex is greater than that of the zinc (Kunchert, *loc. cit.*) the effects in the two cases are not of the same magnitude.

As a result of these considerations all the substances other than sodium cyanide which have been added to brass plating solutions are arbitrarily classified as acid, neutral, or alkaline. It is believed that their influence can be explained by their action as acid, neu-

tral, or alkaline substances upon the above equilibria existing in brass plating solutions.

A set of experiments was carried out with each of the three acid substances, ammonium chloride, sodium hydrogen sulphite, and boric acid. Ammonium chloride and sodium hydrogen sul-

TABLE X.

*Sodium Hydrogen Sulphite.*

| Row | Efficiencies | | | Per-cent of Copper in Deposit | Grams $NaHSO_3$ per Liter | Potential Measurements | | | | | | Character of Deposit |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Anodes | | Cath. | | | With Current | | | Without Current | | | |
| | | | | | | Anodes | | Cath. | Anodes | | Cath. | |
| | Copper | Zinc | | | | Copper | Zinc | | Copper | Zinc | | |
| 1 | 106.9 | 106.8 | 95.2 | 62.6 | 0.0 | 0.864<br>0.876<br>0.880 | 0.970<br>0.967<br>0.951 | 1.390 | 0.952<br>0.964 | 1.038<br>1.049 | 0.984 | Dull Red |
| 2 | 101.6 | 111.7 | 97.8 | 72.3 | 5.0 | 0.865<br>0.868<br>0.873 | 0.907<br>0.917<br>0.912 | 1.358 | 0.942<br>0.945 | .985<br>1.004 | 0.963 | " " |
| 3 | 103.7 | 124.2 | 99.1 | 77.0 | 10.0 | 0.858<br>0.866<br>0.867 | 0.883<br>0.890<br>0.880 | 1.307 | 0.914<br>0.926 | 0.981<br>0.996 | 0.940 | " " |
| 4 | 105.9 | 148.3 | 99.0 | 80.6 | 15.0 | 0.841<br>0.844<br>0.849 | 0.860<br>0.863<br>0.853 | 1.220 | 0.887<br>0.901 | 0.924<br>0.901 | 0.920 | " " |
| 5 | 109.4 | 128.3 | 99.9 | 85.7 | 20.0 | 0.828<br>0.828<br>0.830 | 0.821<br>0.485<br>0.153 | 1.144 | 0.878<br>0.883 | 0.940<br>0.910 | 0.899 | " " |
| 6 | 111.5 | 112.0 | 97.1 | 96.9 | 30.0 | 0.797<br>0.770<br>0.470 | —0.723<br>—0.723<br>—0.723 | 1.082 | 0.837<br>0.843 | 0.899<br>0.901 | 0.875 | " " |

phite were selected because they are the most commonly used acid substances. Boric acid was used because its action can be only that of a weak acid. The results obtained by the addition of variable amounts of each of these substances are recorded in Tables X, XI and XII.

The general influence of the three substances is the same. Sodium hydrogen sulphite produces the greatest effect and ammonium chloride the least. Since sodium hydrogen sulphite is only

slightly acid, the effect observed here seems greater than was to be expected. This point is given further consideration following the discussion of alkaline substances. The potentials in all cases show a regular decrease (Curves 1 and 2, Plates IV and V), which indicates an increase in metal ion concentration. This in-

TABLE XI.

*Ammonium Chloride.*

| Row | Efficiencies | | | Per-cent of Copper in Deposit | Grams $NH_4Cl$ per Liter | Potential Measurements | | | | | | Character of Deposit |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Anodes | | Cath. | | | With Current | | | Without Current | | | |
| | | | | | | Anodes | | Cath. | Anodes | | Cath. | |
| | Copper | Zinc | | | | Copper | Zinc | | Copper | Zinc | | |
| 1 | 103.1 | 87.6 | 97.3 | 65.6 | 0.0 | 0.892<br>0.895<br>0.891 | 0.968<br>0.965<br>0.966 | 1.445 | 0.962<br>0.973 | 1.062<br>1.039 | 0.994 | Dull red |
| 2 | 103.1 | 58.8 | 98.2 | 66.3 | 3.0 | 0.880<br>0.882<br>0.887 | 0.950<br>0.950<br>0.940 | 1.421 | 0.967<br>0.970 | 1.052<br>1.038 | 0.993 | " " |
| 3 | 103.1 | 82.9 | 97.5 | 68.9 | 5.0 | 0.898<br>0.904<br>0.903 | 0.962<br>0.958<br>0.930 | 1.436 | 0.959<br>0.971 | 1.046<br>1.034 | 0.992 | " " |
| 4 | 102.6 | 81.9 | 98.0 | 68.7 | 7.0 | 0.893<br>0.899<br>0.903 | 0.975<br>0.969<br>0.935 | 1.423 | 0.968<br>0.973 | 1.034<br>1.016 | 0.996 | " " |
| 5 | 92.2 | 72.4 | 97.8 | 71.9 | 11.0 | 0.909<br>0.904<br>0.900 | 0.903<br>0.909<br>0.934 | 1.400 | 0.956<br>0.965 | 1.011<br>1.021 | 0.993 | " " |
| 6 | 100.9 | 57.0 | 97.6 | 72.5 | 13.0 | 0.903<br>0.903<br>0.902 | 0.915<br>0.930<br>0.921 | 1.395 | 0.962<br>0.970 | 0.998<br>1.021 | 0.998 | " " |

crease in metal ion concentration is to be expected from the above conclusions regarding the influence of acid substances upon the equilibria which exist in the solution. The decrease in cathode polarization (Curve 3, Plates IV and V) is accompanied by the customary increase in the percentage of copper in the deposit (Curve 4, Plates IV and V). It is concluded from these results that no advantage is to be gained by the addition of any of these substances to a solution of the composition here used. A solution

of lower copper content, which normally gives a deposit too low in copper, may be made to give a deposit of the desired copper content by the addition of acid substances.

TABLE XII.

*Boric Acid.*

| Row | Efficiencies | | | Per-cent of Copper in Deposit | Grams of Boric Acid per Liter | Potential Measurements | | | | | | Character of Deposit |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Anodes | | Cath. | | | With Current | | | Without Current | | | |
| | | | | | | Anodes | | Cath. | Anodes | | Cath. | |
| | Copper | Zinc | | | | Copper | Zinc | | Copper | Zinc | | |
| 1 | 103.4 | 94.6 | 97.8 | 65.7 | 0.0 | 0.831<br>0.854<br>0.857 | 0.918<br>0.925<br>0.975 | 1.467 | 0.895<br>0.925 | 1.072<br>1.063 | 0.997 | Dull red |
| 2 | 102.2 | 111.3 | 98.4 | 67.4 | 2.0 | 0.846<br>0.849<br>0.850 | 0.940<br>0.929<br>0.926 | 1.468 | 0.924<br>0.943 | 1.014<br>1.038 | 0.981 | " " |
| 3 | 99.8 | 115.9 | 99.2 | 67.7 | 4.0 | 0.856<br>0.856<br>0.857 | 0.920<br>0.916<br>0.920 | 1.460 | 0.930<br>0.943 | 1.028<br>1.023 | 0.973 | " " |
| 4 | 99.2 | 117.8 | 99.2 | 71.2 | 8.0 | 0.838<br>0.838<br>0.841 | 0.924<br>0.916<br>0.910 | 1.458 | 0.917<br>0.926 | 1.017<br>1.000 | 0.959 | " " |
| 5 | 100.6 | 111.8 | 99.4 | 71.4 | 15.0 | 0.830<br>0.830<br>0.833 | 0.884<br>0.875<br>0.882 | 1.432 | 0.906<br>0.921 | 0.994<br>1.000 | 0.950 | " " |
| 6 | 101.2 | 115.9 | 98.8 | 73.7 | 20.0 | 0.827<br>0.835<br>0.841 | 0.892<br>0.885<br>0.881 | 1.412 | 0.886<br>0.908 | 0.977<br>0.993 | 0.938 | " " |
| 7 | 98.3 | 110.3 | 100.6 | 78.6 | 25.0 | 0.845<br>0.845<br>0.845 | 0.829<br>0.832<br>0.825 | 1.354 | 0.892<br>0.910 | 0.981<br>0.975 | 0.931 | " " |

*Alkaline Substances.*

The alkaline substances first used were ammonium hydroxide and sodium carbonate. The results produced by the addition of variable amounts of each of these substances to the original solution are recorded in Tables XIII and XIV.

The increase in potential of both copper and zinc (Curves 1 and 2, Plates VI and VII) shows that the metal ion concentra-

tions decrease with increase in the alkalinity of the solution. This decrease in metal ion concentration produces an increase in cathode polarization (Curve 3, Plates VI and VII) and thus a decrease in the percentage of copper in the deposit (Curve 4,

TABLE XIII.

*Ammonium Hydroxide.*

| Row | Efficiencies | | | Per-cent of Copper in Deposit | Equivalent of $NH_4OH$ per Liter | Potential Measurements | | | | | | Character of Deposit |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Anodes | | Cath. | | | With Current | | | Without Current | | | |
| | | | | | | Anodes | | Cath. | Anodes | | Cath. | |
| | Copper | Zinc | | | | Copper | Zinc | | Copper | Zinc | | |
| 1 | 103.8 | 95.5 | 99.3 | 63.9 | 0.0 | 0.830<br>0.846<br>0.842 | .975<br>.972<br>.992 | 1.425<br>1.435 | 0.907<br>0.935 | 1.090<br>1.028 | 1.005 | Dull red |
| 2 | 103.5 | 93.4 | 98.4 | 58.0 | 0.15 | 0.918<br>0.919<br>0.916 | 1.019<br>1.011<br>1.007 | 1.460<br>1.475 | 0.968<br>1.005 | 1.127<br>1.109 | 1.021 | Bright yellow |
| 3 | 103.3 | 95.4 | 99.6 | 55.3 | 0.30 | 0.894<br>0.910<br>0.916 | 1.045<br>1.040<br>1.056 | 1.468<br>1.475 | 0.970<br>1.008 | 1.097<br>1.121 | 0.986 | " " |
| 4 | 103.2 | 95.8 | 98.9 | 54.6 | 0.45 | 0.950<br>0.945<br>0.951 | 0.994<br>1.018<br>1.005 | 1.481<br>1.485 | 1.020<br>1.031 | 1.170<br>1.135 | 1.011 | " " |
| 5 | 101.9 | 128.9 | 99.4 | 53.3 | 0.60 | 0.936<br>0.927<br>0.924 | 1.020<br>1.001<br>0.965 | 1.504<br>1.500 | 1.021<br>1.028 | 1.140<br>1.198 | 1.003 | " " |
| 6 | 102.2 | 95.7 | 99.3 | 54.6 | 0.75 | 0.951<br>0.951<br>0.949 | 1.055<br>1.023<br>1.014 | 1.484<br>1.483 | 1.028<br>1.032 | 1.160<br>1.172 | 0.991 | " " |
| 7 | 104.6 | 101.3 | 99.3 | 53.0 | 1.05 | 0.979<br>0.975<br>0.948 | 1.084<br>1.090<br>1.041 | 1.499<br>1.474 | 1.059<br>1.047 | 1.219<br>1.215 | 1.056 | " " |

Plates VI and VII). As is to be expected, the influence of these substances is opposite to that of acid substances. Ammonium hydroxide has a greater effect than sodium carbonate, but the effect in both cases compared with that of acid substances is small.

These substances have a pronounced effect on the nature of the deposit. A bright-yellow deposit is obtained in both cases. As the percentage of copper in the deposit decreases the brass changes

from a dull red to a bright yellow at about 58 percent of copper, and remains bright yellow throughout the determinations. From these results it appears desirable to add a small quantity of an alkaline substance to the solution because of the favorable influence it has on the nature of the deposit.

It was pointed out above that sodium hydrogen sulphite produces a greater effect than is to be expected from its acidity. This

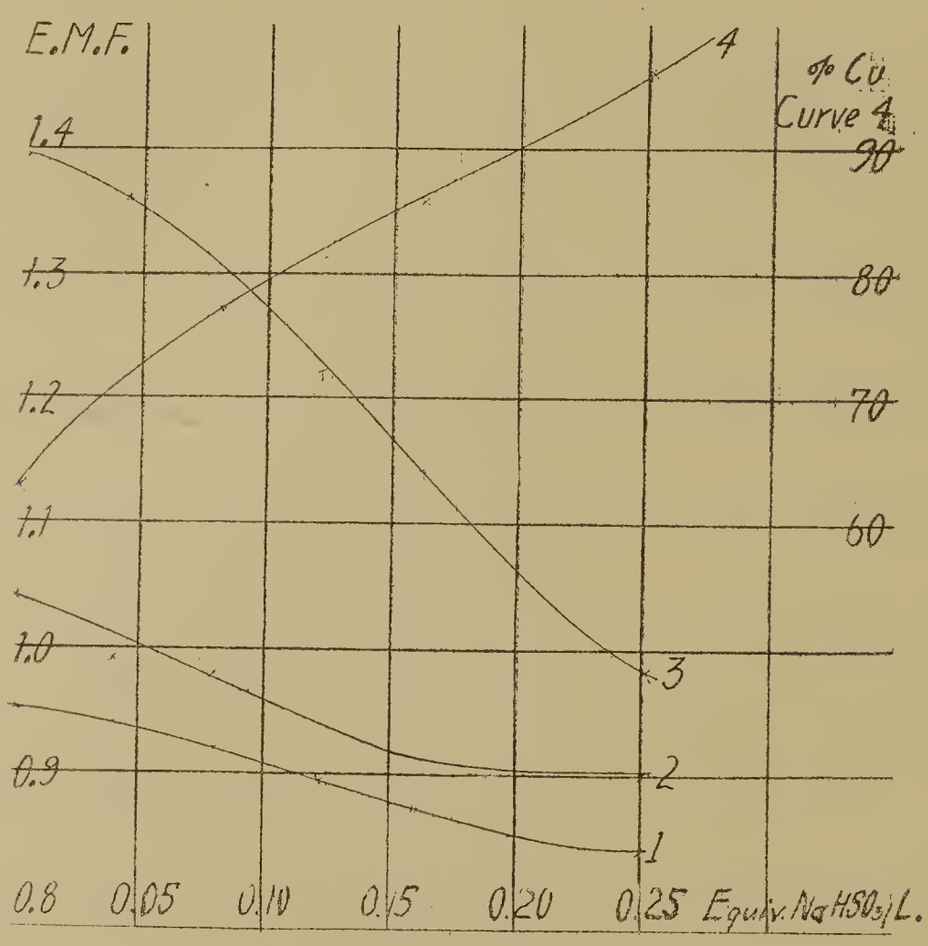


PLATE IV. Influence of Sodium Hydrogen Sulphite.

1. Potential of copper anode without current.
2. Potential of zinc anode without current.
3. Potential of cathode with current.
4. Percentage of copper in the deposit.

suggested that it might have some influence other than that of an acid substance. It was thought possible that it might exert a reducing effect on the solution and thus influence the metal ion concentration. In order to obtain more information on this point two series of experiments were performed; one, by the addition of variable amounts of sodium hydrogen sulphate, and the other, by the addition of variable amounts of sodium sulphite. Sodium sulphite was used to obtain the influence of the sulphite ion in

the absence of an acid substance. Sodium hydrogen sulphate was used to obtain the influence of the hydrogen ion in the absence of the sulphite ion. The results obtained by the addition of these substances to the original solution are given in Tables XV and XVI.

TABLE XIV.

*Sodium Carbonate.*

| Row | Efficiencies | | | Per-cent of Copper in Deposit | Equivalent of $Na_2CO_3$ per Liter | Potential Measurements | | | | | | Character of Deposit |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Anodes | | Cath. | | | With Current | | | Without Current | | | |
| | | | | | | Anodes | | Cath. | Anodes | | Cath. | |
| | Copper | Zinc | | | | Copper | Zinc | | Copper | Zinc | | |
| 1 | 103.0 | 89.2 | 98.2 | 64.8 | 0.0 | 0.852<br>0.853<br>0.849 | 0.941<br>0.937<br>0.931 | 1.440 | 0.966<br>0.959 | 1.091<br>1.064 | 0.989 | Dull red |
| 2 | 102.5 | 88.4 | 98.2 | 57.0 | 0.094 | 0.857<br>0.869<br>0.878 | 0.930<br>0.998<br>0.988 | 1.440 | 0.953<br>0.969 | 1.036<br>1.079 | 0.999 | " reddish yellow |
| 3 | 102.8 | 97.6 | 99.4 | 57.62 | 0.188 | 0.866<br>0.874<br>0.873 | 0.993<br>0.957<br>0.961 | 1.430 | 0.966<br>0.971 | 1.066<br>1.079 | 0.998 | " yellow |
| 4 | 103.2 | 94.8 | 99.4 | 58.2 | 0.282 | 0.873<br>0.869<br>0.869 | 0.952<br>0.958<br>0.962 | 1.439 | 0.968<br>0.972 | 1.088<br>1.107 | 0.998 | Bright " |
| 5 | 102.0 | 94.7 | 99.3 | 58.0 | 0.376 | 0.869<br>0.866<br>0.864 | 0.981<br>0.972<br>0.976 | 1.430 | 0.984<br>0.973 | 1.094<br>1.133 | 1.000 | " " |
| 6 | 101.5 | 89.6 | 98.8 | 56.8 | 0.564 | 0.861<br>0.855<br>0.855 | 0.995<br>0.979<br>0.987 | 1.451 | 0.996<br>0.979 | 1.177<br>1.099 | 1.007 | " " |
| 7 | 101.6 | 91.7 | 98.7 | 55.6 | 0.752 | 0.861<br>0.856<br>0.853 | 1.015<br>1.015<br>0.997 | 1.454 | 1.001<br>0.998 | 1.154<br>1.125 | 1.004 | " " |

The changes produced by sodium sulphite are slight. The only observed effect is that of an extremely weak base. This indicates that the sulphite ion has no specific influence. Sodium hydrogen sulphate has approximately the same effect as sodium hydrogen sulphite. One would expect sodium hydrogen sulphate to have the greater effect, since in a pure solution of sodium hydrogen sulphate the hydrogen ion concentration is distinctly greater than

in a pure solution of sodium hydrogen sulphite. Even though the influence of sodium hydrogen sulphite is more pronounced than can be accounted for by the concentration of hydrogen ions in a pure solution, nevertheless it appears to have no influence other than that of an acid substance. In the preparation of the original solution, however, a quantity of sodium hydrogen sulphite equivalent to the copper is beneficial in that it prevents the loss of

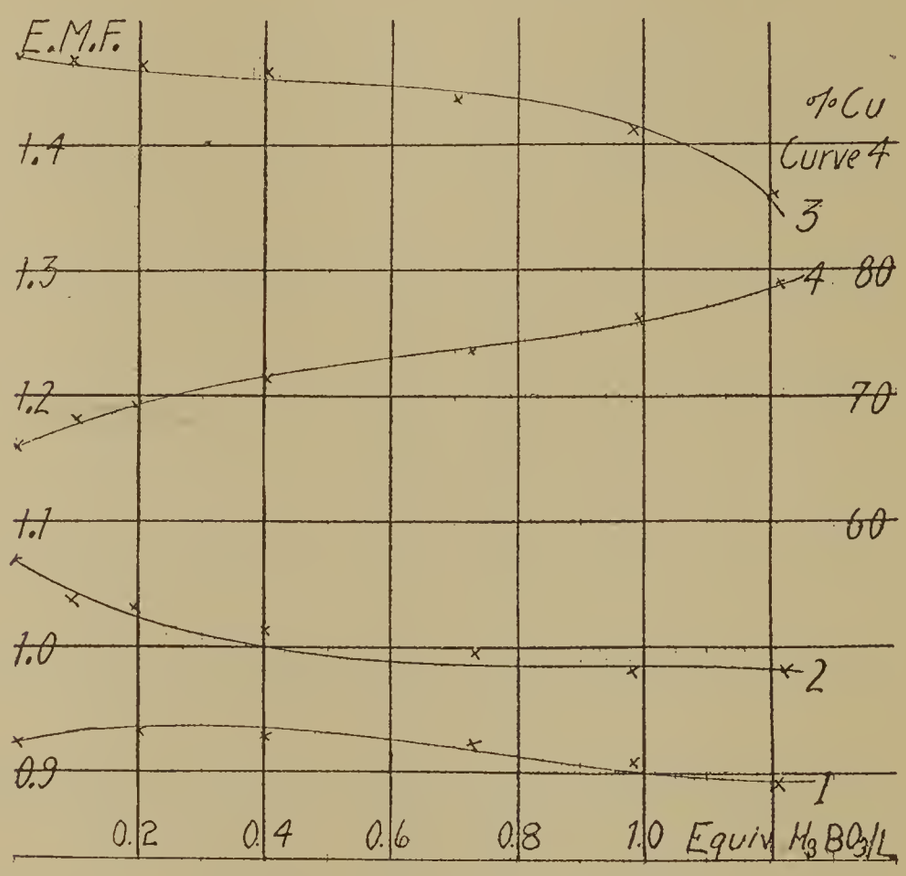


PLATE V. Influence of Boric Acid.

1. Potential of copper anode without current.
2. Potential of zinc anode without current.
3. Potential of cathode with current.
4. Percentage of copper in the deposit.

cyanide (W. D. Bancroft, Jour. Phys. Chem. (1905), **9**, 277).

It is concluded from these results that the changes produced by any of the acid substances may be obtained by the addition of a weak acid, and that ammonium hydroxide may be used in place of any of the alkaline substances.

### *Neutral Substances.*

In the preparation of brass plating solutions the metal content is obtained from the chlorides, nitrates, sulphates, acetates, carbonates, or oxides of copper and zinc. When sodium cyanide is

added to solutions of these salts the sodium salts of these acids are formed in the solution. This is equivalent, in the case of acetates, carbonates, or oxides, to the addition of alkaline substances; and in the case of chlorides, nitrates, or sulphates, to the

TABLE XV.

*Sodium Sulphite.*

| Row | Efficiencies | | | Per-cent of Copper in Deposit | Grams $Na_2SO_3$ per Liter | Potential Measurements | | | | | | Character of Deposit |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Anodes | | Cath. | | | With Current | | | Without Current | | | |
| | | | | | | Anodes | | Cath. | Anodes | | Cath. | |
| | Copper | Zinc | | | | Copper | Zinc | | Copper | Zinc | | |
| 1 | 103.0 | 92.0 | 90.2 | 63.7 | 0.0 | 0.857<br>0.858<br>0.863 | 0.903<br>0.958<br>0.946 | 1.493 | 0.916<br>0.944 | 1.021<br>1.037 | 0.995 | Dull red yellow |
| 2 | 102.5 | 90.0 | 97.0 | 60.5 | 5.0 | 0.849<br>0.851<br>0.851 | 0.953<br>0.943<br>0.932 | 1.504 | 0.934<br>0.949 | 1.053<br>1.051 | 0.996 | Slightly dull yellow |
| 3 | 102.3 | 97.6 | 96.4 | 62.7 | 10.0 | 0.842<br>0.847<br>0.844 | 0.985<br>0.973<br>0.980 | 1.498 | 0.943<br>0.962 | 1.036<br>1.042 | 0.996 | " " " |
| 4 | 101.7 | 95.7 | 97.5 | 61.9 | 15.0 | 0.868<br>0.870<br>0.869 | 0.968<br>0.964<br>0.976 | 1.493 | 0.953<br>0.960 | 1.030<br>1.038 | 0.991 | " " " |
| 5 | 100.9 | 114.5 | 95.4 | 61.1 | 20.0 | 0.833<br>0.850<br>0.851 | 0.921<br>0.910<br>0.910 | 1.500 | 0.934<br>0.945 | 1.033<br>1.043 | 0.982 | " " " |
| 6 | 100.3 | 106.0 | 96.0 | 60.8 | 25.0 | 0.851<br>0.857<br>0.859 | 0.935<br>0.936<br>0.922 | 1.509 | 0.940<br>0.951 | 1.027<br>1.049 | 0.980 | " " " |
| 7 | 100.7 | 92.5 | 96.2 | 60.9 | 30.0 | 0.853<br>0.857<br>0.855 | 0.939<br>0.928<br>0.917 | 1.520 | 0.930<br>0.935 | 1.049<br>1.069 | 0.979 | " " " |

addition of neutral substances. It was considered advisable, therefore, to determine whether these neutral salts influenced the action of the solution. To do this a series of experiments was performed in which various quantities of sodium sulphate were added to the original solution. The results obtained by such additions are given in Table XVII.

The changes produced are so slight that they are within the limits of experimental error. These results are to be expected since it appears, from the above discussion of the equilibria existing in cyanide solutions, that only substances of an acid or alkaline nature can influence the metal ion concentration.

TABLE XVI.

*Sodium Acid Sulphate.*

| Row | Efficiencies | | | Per-cent of Copper in Deposit | Grams $NaHSO_4$ per Liter | Potential Measurements | | | | | | Character of Deposit |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Anodes | | Cath. | | | With Current | | | Without Current | | | |
| | | | | | | Anodes | | Cath. | Anodes | | Cath. | |
| | Copper | Zinc | | | | Copper | Zinc | | Copper | Zinc | | |
| 1 | 102.6 | 104.6 | 98.3 | 62.6 | 0.0 | 0.874<br>0.869<br>0.866 | 0.932<br>0.936<br>0.906 | 1.496<br>1.508<br>1.508 | 0.944<br>0.945 | 1.025<br>1.020 | 0.997 | Dull red-yellow |
| 2 | 103.2 | 118.8 | 98.3 | 63.7 | 2.5 | 0.854<br>0.853<br>0.851 | 0.943<br>0.915<br>0.919 | 1.480<br>1.485<br>1.479 | 0.897<br>0.926 | 1.021<br>1.035 | 0.982 | Dull red |
| 3 | 102.3 | 107.5 | 98.3 | 69.0 | 5.0 | 0.857<br>0.859<br>0.856 | 0.905<br>0.900<br>0.900 | 1.491<br>1.474<br>1.471 | 0.902<br>0.931 | 1.018<br>1.022 | 0.971 | " " |
| 4 | 102.7 | 110.9 | 98.2 | 75.1 | 10.0 | 0.866<br>0.867<br>0.862 | 0.892<br>0.898<br>0.888 | 1.431<br>1.438<br>1.433 | 0.889<br>0.916 | 0.972<br>0.989 | 0.953 | " " |
| 5 | 103.1 | 116.9 | 99.2 | 79.1 | 15.0 | 0.852<br>0.851<br>0.851 | 0.875<br>0.884<br>0.888 | 1.390<br>1.387<br>1.385 | 0.882<br>0.900 | 0.951<br>0.969 | 0.930 | " " |
| 6 | 101.3 | 109.7 | 99.5 | 80.1 | 20.0 | 0.831<br>0.817<br>0.809 | 0.853<br>0.869<br>0.876 | 1.371<br>1.361<br>1.374 | 0.861<br>0.885 | 0.933<br>0.976 | 0.912 | " " |

*Brass as Anodes.*

In all of this work copper and zinc anodes were used to make possible the study of various factors by the measurement of single electrode potentials. This method of maintaining the metal content of the solution, however, is unsatisfactory. The zinc becomes covered with a layer of copper which interferes with its normal behavior. During electrolysis this deposit drops from the electrode and collects as a sediment. Such action results in an uncontrollable change in the metal content of the solution.

In an earlier experiment (see Table V) a solution was electrolyzed for a period of eighty-eight hours. During this time the current through each anode was so regulated that the metals dissolved and deposited in the same ratio. At the end of this time analysis showed that the solution had increased in metal content, probably due to the solvent action of the cyanide, but the zinc had increased relatively more than the copper. This reduced the ratio

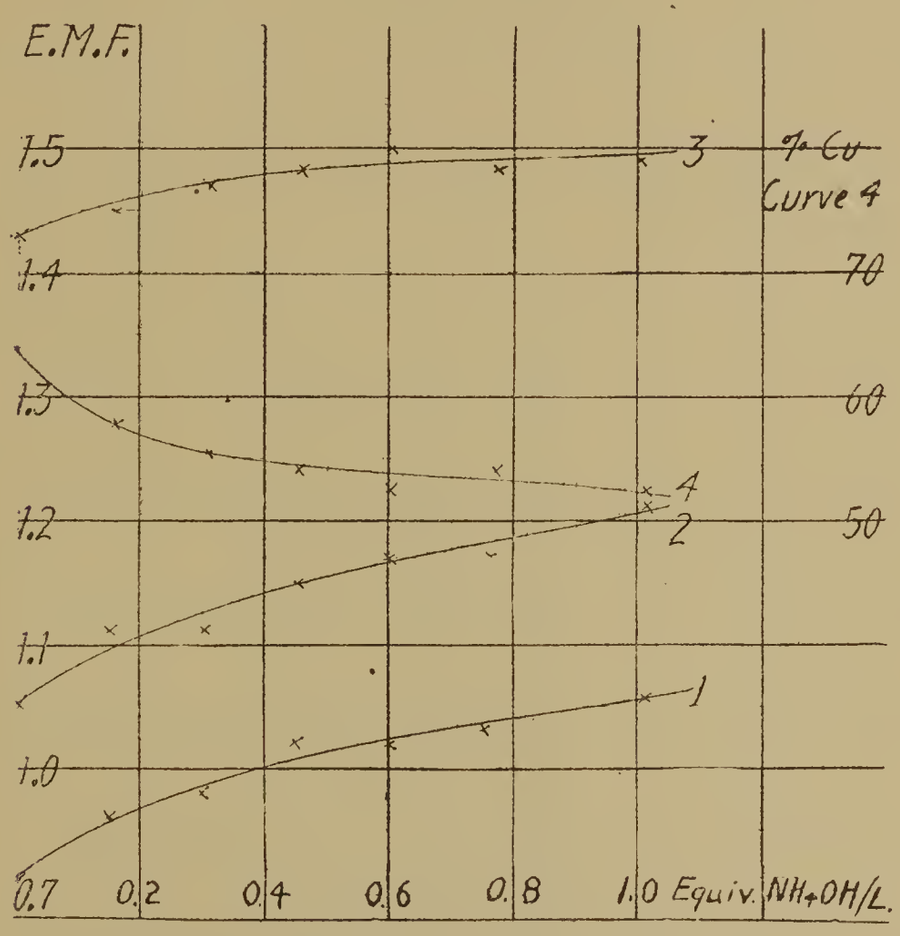


PLATE VI. Influence of Ammonium Hydroxide.
1. Potential of copper anode without current.
2. Potential of zinc anode without current.
3. Potential of cathode with current.
4. Percentage of copper in the deposit.

of copper to zinc in the solution and consequently reduced the percentage of copper in the deposit.

Experiments were consequently performed to determine the anodic behavior of brass. For this purpose brass anodes, the composition of which varied from 62.5 percent to 85.0 percent copper, were cast and their efficiencies of corrosion determined. The value recorded for the efficiency in each case is the average of ten determinations. The results obtained are given in Table XVIII.

The brass anodes maintain their original bright yellow color and no sediment forms during the electrolysis. The surface of the electrodes remains smooth and there is apparently no tendency for zinc to dissolve in preference to copper, as might be expected from the difference in the solution pressures of these metals; in

TABLE XVII.

*Sodium Sulphate.*

| Row | Efficiencies | | | Per-cent of Copper in Deposit | Grams $Na_2SO_4$ per Liter | Potential Measurements | | | | | | Character of Deposit |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Anodes | | Cath. | | | With Current | | | Without Current | | | |
| | | | | | | Anodes | | Cath. | Anodes | | Cath. | |
| | Copper | Zinc | | | | Copper | Zinc | | Copper | Zinc | | |
| 1 | 102.3 | 95.5 | 91.5 | 61.4 | 0.0 | 0.860<br>0.854<br>0.851 | 0.941<br>0.947<br>0.950 | 1.503<br>1.502<br>1.501 | 0.906<br>0.932 | 1.035<br>1.042 | 0.991 | Dull red-yellow |
| 2 | 99.4 | 98.5 | 96.1 | 61.7 | 5.0 | 0.837<br>0.840<br>0.840 | 0.944<br>0.948<br>0.915 | 1.500<br>1.493<br>1.491 | 0.911<br>0.929 | 1.038<br>1.042 | 0.983 | Slightly dull yellow |
| 3 | 102.7 | 93.0 | 97.3 | 63.8 | 15.0 | 0.831<br>0.844<br>0.845 | 0.935<br>0.933<br>0.900 | 1.478<br>1.497<br>1.493 | 0.893<br>0.935 | 1.042<br>1.048 | 0.980 | " " |
| 4 | 102.3 | 107.3 | 97.6 | 65.3 | 25.0 | 0.848<br>0.847<br>0.838 | 0.931<br>0.950<br>0.940 | 1.481<br>1.479<br>1.479 | 0.904<br>0.937 | 1.052<br>1.029 | 0.976 | " " |
| 5 | 102.6 | 103.5 | 98.9 | 66.2 | 35.0 | 0.840<br>0.845<br>0.846 | 0.934<br>0.939<br>0.943 | 1.453<br>1.454<br>1.455 | 0.906<br>0.938 | 1.048<br>1.047 | 0.979 | " " |
| 6 | 99.8 | 118.6 | 99.1 | 65.3 | 45.0 | 0.825<br>0.826<br>0.828 | 0.924<br>0.925<br>0.925 | 1.475<br>1.476<br>1.466 | 0.908<br>0.930 | 1.025<br>1.035 | 0.972 | " " |

other words, brasses of the composition here used dissolve uniformly as such. The efficiency of corrosion is in all cases about the same as that of copper.

*Single Potential of Brass.*

In all the tables in which the single potentials of copper and zinc are given it is observed that the potentials of zinc are approximately 0.1 volt or more greater than those of copper. According

to the theory of electro-deposition it is impossible for two metals to be deposited simultaneously when their potentials differ by this amount. Some other highly influential factor, which has not yet been considered, must consequently be active here.

It is a well-known fact that strongly electro-negative metals can, under certain conditions, be deposited from solutions at potentials much lower than their equilibrium potentials. For example, sodium may be deposited from an aqueous solution of its salts, provided mercury be used as cathode. This is explained

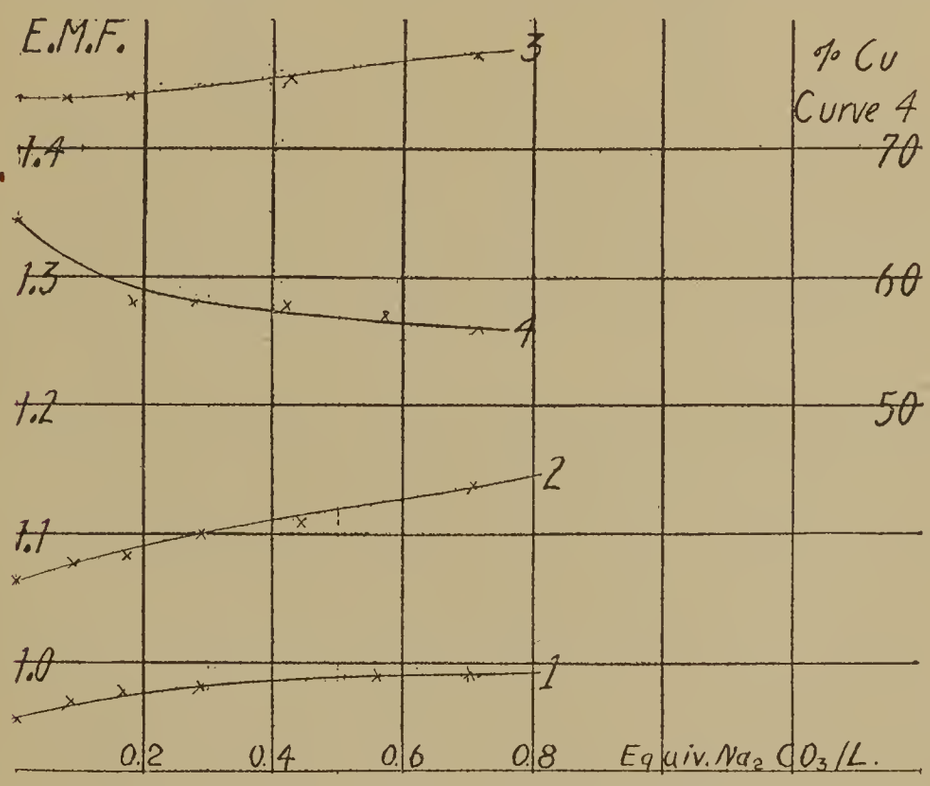


PLATE VII. Influence of Sodium Carbonate.

1. Potential of copper anode without current.
2. Potential of zinc anode without current.
3. Potential of cathode with current.
4. Percentage of copper in the deposit.

by assuming that sodium alloys with the mercury, and the resulting alloy shows a low electrolytic solution pressure for sodium. It appears that some similar action must take place in the deposition of brass from cyanide solutions.

It is known that zinc and copper do form alloys. The potentials which such alloys show depend upon their nature and composition. A. J. Allmand (Principles of Applied Electrochemistry, p. 136) pointed out that the potentials of those alloys which are simply solid solutions of one metal in another vary continuously with composition from the potential of one metal to that of the

other. In case the two metals form a compound, it shows a characteristic potential which may lie between the potentials of the two metals or may even exceed the potentials of either. If the alloy consists of a mixture of two or more constituents the potential which the alloy shows is that of the more electro-negative constituent.

TABLE XVIII.

| Percentage of Copper | Efficiency of Corrosion |
|---|---|
| 62.3 | 104.8 |
| 66.2 | 105.2 |
| 70.7 | 103.8 |
| 76.0 | 103.4 |
| 81.4 | 103.2 |
| 85.0 | 104.0 |

It seems entirely possible that a thin film of copper is first deposited, and this greatly decreases the potential necessary for the deposition of zinc. This depolarization may be so great that the simultaneous deposition of the two metals takes place from solutions in which their single potentials are not the same. The two metals thus deposited form an alloy, the potential and other properties of which depend upon its nature and composition.

In order to obtain some information on the potentials of brasses formed in this way, ten samples of brass, the composition of which ranged from 37.6 to 82.0 percent of copper, were prepared by varying the acidity of the stock solution. The potentials of these brasses were then measured in the original solution and the data recorded in Table XIX.

TABLE XIX.

| Percent of Copper | Potential |
|---|---|
| 37.6 | 1.005 |
| 56.7 | 0.925 |
| 62.3 | 0.928 |
| 66.0 | 0.917 |
| 69.5 | 0.978 |
| 72.2 | 0.979 |
| 79.1 | 0.984 |
| 82.0 | 0.990 |
| Pure Copper | 0.914 |
| Pure Zinc | 1.080 |

It is seen from this table that the potential decreases to a minimum with increase in copper to 66.0 percent and then increases. Since a brass deposit which contains 66.0 percent of copper is the

most satisfactory, there may be some relation between this low potential and the nature of the deposit. The potentials of these brasses lie between the single potentials of copper and zinc in the same solution and most of them are much nearer the potential of copper than the potential of zinc. These results lead one to believe that the alloys formed by electro-deposition are solid solutions of a compound of copper and zinc in copper. Further experiments of this kind would doubtless afford valuable information regarding the exact nature of copper-zinc alloys.

*Summary.*

As a result of this work the following statements may be made:

(1). Increase in the ratio of copper to zinc in the solution increases the percentage of copper in the deposit. A solution in which the ratio of copper to zinc is 4.2 gives a deposit of about 65 percent copper (ratio 1.9).

(2) Solutions of high metal content are more satisfactory than dilute solutions. A solution containing thirty-five grams of metal per liter, in the above ratio, gives satisfactory deposits.

(3) Increase in temperature decreases cathode polarization and consequently increases the percentage of copper in the deposit.

(4) Increase in current density produces a gradual decrease in the percentage of copper in the deposit. At current densities greater than 0.3 ampere per sq. dm., the deposit becomes granular, non-adherent, and dull in color.

(5) Increase in free cyanide does not increase anode efficiency, but does decrease cathode efficiency. Its influence on the percentage of copper in the deposit is variable.

(6) Slightly acid substances increase the percentage of copper in the deposit. A weak acid may be used in place of any of the acid substances that have been recommended.

(7) Slightly alkaline substances decrease the percentage of copper in the deposit. The presence of slightly alkaline substances is beneficial in that it improves the appearance of the deposit.

(8) Neutral substances have no influence on the deportment of the cyanide brass plating solution.

(9) Brasses which vary in composition from 62.3 to 85.0 percent of copper dissolve as such anodically. The efficiency of corrosion is about the same as that of copper.

(10) Decided depolarization of zinc by copper takes place and makes possible the deposition of brass from solutions in which the potentials of the two metals are not equal.

(11) Electro-deposited brasses which vary in composition from 37.6 to 82.0 percent copper give nearly the same potentials in a plating solution. These potentials are nearer to that of copper than to that of zinc.

The writer wishes to express his sincere appreciation for the kind advice and instruction during the progress of this work and for the criticism of the thesis rendered by Dr. Alfred L. Ferguson, at whose suggestion and under whose supervision this investigation was carried out.

To Professor S. Lawrence Bigelow, for his most valuable criticism of this thesis, sincere thanks are also due.

*Chemical Laboratory,*
*University of Michigan.*

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## DISCUSSION.

Wm. Blum[1]: If, as indicated in the remarks of Dr. Ferguson, the solutions were prepared by the addition of sodium cyanide to sulphates of copper and zinc, the statement on p. 174 that no constituents other than complex cyanides were present is misleading, since an equivalent of sodium sulphate must also have been formed. It is therefore at least not certain that the conclusions reached would apply also to baths containing only the double cyanides, made by the solution in sodium cyanide of the cyanides of zinc and copper, such as are actually employed in the preparation of electroplating baths.

One of the most uncertain analytical control methods in brass

[1] Chemist, Bureau of Standards, Washington, D. C.

(or cyanide copper) plating, is the determination of the "free" cyanide or even of the total cyanide content, since the results obtained by direct titration are at best only an approximation. This subject is greatly in need of study. However it would be at least interesting if Dr. Ferguson could include some figures even if only approximate, for the free cyanide content of the solutions as prepared, after the boiling and after operation for considerable periods, in order to throw light upon the changes which take place during operation. Qualitatively, such changes might be followed by the determination of carbonates, which are the principal final decomposition product of cyanides.

It should be noted that all the deposits were made upon platinum, in order to facilitate their subsequent analysis. It would be desirable to analyze similar deposits made upon iron, to determine whether the base metal has an appreciable influence, as may well be the case when, as Dr. Ferguson points out, polarization phenomena are involved.

The universities are to be commended for undertaking such studies upon electrodeposition and its commercial applications, since there is today so great a need and demand for information in this field. In the conduct of such investigations every effort should be made to correlate the work and results with actual plating practice; in short we should start out with and add to the information which is now possessed by the practical platers. There is all too great a tendency among chemists to lay stress upon those statements regarding electrodeposition which are found in the scientific literature or in hand books on plating, and to ignore that large fund of valuable experience possessed by practical platers, who usually are unable to express their information in scientific language. Brass plating, for example, is not a laboratory operation, but a process conducted in 500 or more plants in the United States. It is carried out successfully in the sense that most of the time the men can "deliver the goods". It is not carried out successfully in the sense that the operators know or understand its underlying principles, or are able to get uniformly satisfactory results, or to at once remedy difficulties that may arise. Such knowledge can be gained only through exhaustive and fundamental scientific studies, but much time can be saved by taking into account what the plater has already learned.

Formerly such co-ordination was no doubt hindered by the secretiveness of platers. This condition has passed however, and I can state from personal experience that the American Electro-Platers' Society (which is a sort of younger brother of this organization) will co-operate gladly in such work. I would therefore suggest that persons desiring to study electroplating problems should consult with platers in their vicinity, and if possible affiliate with one of the numerous branches of the American Electro-Platers' Society and exchange views with them.

The Bureau of Standards is trying to do some work in this field, but with present resources it is not possible for us to meet the great need. The co-operation of educational institutions is greatly needed. If in any way we can serve as a clearing house between chemists and platers, and bring them into closer association, we will be glad to do so.

W. R. Mott[2]: The point has been brought out that there is a possibility of a special alloy between copper and zinc that lowers the zinc potential. In a paper by Dr. H. E. Patton and myself[3] it was proved that low potentials of the zinc with copper were due to the high heats of formation of their alloys. (At the percentage $Zn_2Cu$, that is 66 atoms percent of zinc, the heat of formation of the alloy is a maximum.) This large heat of formation is the fundamental reason why you have a readjustment of the energy, which must be of single potential voltage because Faraday's law fixes the current component of the energy.

G. B. Hogaboom[4]: Dr. Blum has spoken of the technical side and my being a commercial plater compels me to take the standpoint of the application of brass plating to commercial needs. I agree thoroughly with Dr. Blum that it would be far better to obtain a formula for a brass solution from a practical plater who operates a brass solution daily rather than to go to the published literature. Not one of you would think of going to something published 50 years ago for methods of analysis, neither should anyone investigating electro-plating problems depend upon formulae that are given in text books twenty, thirty or fifty years old.

[2] National Carbon Co., Cleveland, Ohio.

[3] Single Potentials of Zinc in Aqueous Solution, Trans. Am. Electrochem. Soc. (1903), 3, 342.

[4] Research Electroplater, Scovill Mfg. Co., Waterbury, Conn.

Dr. Ferguson has not told the entire story in his paper, and some of the results given are not justified in the light of what has been brought out in the discussion. If the brass solution had been analyzed after boiling, he would have found that some of the sodium cyanide had been decomposed into sodium carbonate. If he had added sodium carbonate to the solution when freshly made the same result would have been had as obtained after boiling.

He states that he used cyanide of sodium and a cyanide of metals in making his solution, yet admits that the sulphates of copper and zinc were used. In such a case sodium sulphate would be present and would materially affect the anode and cathode efficiency, and therefore, the figures given for those anode and cathode efficiencies cannot be relied upon unless it is taken in consideration that the sulphate salts were present.

There should be a definition made of the word "corrosion", or rather an explanation of what "corrosion" means. To corrode iron one expects an oxide of iron. To corrode lead by electrolysis the oxide of lead is obtained. In both of these corrosions the materials obtained are oxides of the metals, and in electroplating this is not required. The corrosion of anodes really means how much of the metal goes into the solution and replaces that which has been drawn from the solution and is deposited upon the cathode. The sludge which is formed on the anode and falls to the bottom of the tank is useless, and the loss of metal from such a corrosion should not be considered as anode efficiency.

The deposits from brass solutions are judged more by their color than by their composition. There is a distinct difference in color between a cast or rolled brass and an electrolytically deposited brass. It will be found that an electrolytic brass containing approximately 80 percent copper and 20 percent zinc has a color, upon finishing, almost identical with a rolled brass of 64 percent copper and 36 percent zinc. Color is a prime object of the brass electro-platers and when investigations are made of brass solutions this should be kept in mind.

I wish to state that members of the American Electro-Platers' Society stand ready and willing to offer any assistance that they can to any investigator of electro-plating problems, and in my

opinion it would be far better for any investigator to go to a practical plater or write to several of them, and learn their methods and obtain samples of their work and some of their solutions for analysis rather than to depend upon what has been published in text books. We stand ready to give the members of the Electrochemical Society all the service possible, and it is up to them to ask. It is only by such co-operation that the technical and practical men can be brought together and bring about results that will be commercially useful.

A. L. Ferguson *(Communicated)*: Both Dr. Blum and Mr. Hogaboom have pointed out that the original solution contained some sodium sulfate resulting from the reaction of the sulfates of copper and zinc on sodium cyanide, and the question was raised whether the baths with this sodium sulfate present might behave differently from what they would had the sodium sulfate not been present. It would undoubtedly have been better to have avoided this difficulty by using the cyanides of the metals; but I feel confident that the sodium sulfate present introduced no errors, since from theoretical considerations it should not and it was shown experimentally that further additions of sodium sulfate even up to 45 grams per liter produced no measurable changes in the properties of the bath.

In the discussions by Dr. Blum and Mr. Hogaboom much emphasis was laid on the topic of co-operation between the practical platers and the research workers in our educational institutions, and I feel that it was insinuated that such co-operation was not sought in the present work. If such an opinion exists, I sincerely hope I may correct it, as the principle of co-operation between the industrial world and the research laboratories is one concerning which I have a strong feeling. The movement toward such co-operation meets most heartily with my approval.

I fully recognized that it would be highly desirable to make use of the large fund of valuable information which has been accumulated by the practical platers who have been able to "deliver the goods". I hoped to secure samples of a large number of plating solutions which were giving satisfaction and also any which were not, together with samples of brass from each. With these as a starting point, I felt we might carry out some experiments which

would develop the theories underlying the behavior of brass plating baths. With this object in mind, I became affiliated with the Detroit branch of the American Electro-Platers' Society. I attended several of their meetings and became much interested in the work these men are doing. I admire and envy the man who can go into the factory and get results. I was given an opportunity to tell them what we planned to do and asked for their co-operation and specified the nature of co-operation desired.

The editor of the monthly publication of the American Electro-Platers' Society very kindly gave considerable prominence in that publication to a general request which I sent out for samples of brass-plating solutions and brasses obtained from them. I wrote to Mr. Hogaboom describing what we intended to do and asked for suggestions and samples.

In response to these efforts at co-operation we received not one formula, sample of solution, or sample of brass. Our only recourse then was to consult the recent literature and supposed authorities. No one regrets that this was the situation more than myself.

It is one thing to state that you approve of co-operation and quite another to assume the responsibility of actively responding to an appeal for such co-operation when it is sent out.

*A paper presented at the Thirty-eighth General Meeting of the American Electrochemical Society, in Cleveland, October 2, 1920, President Landis in the Chair.*

# ELECTROLYTIC PRODUCTION OF HYDROGEN PEROXIDE.[1]

By F. W. Skirrow[2] and E. R. Stein.[2]

Abstract.

The literature is briefly abstracted, followed by results of experimental decomposition of sulphuric acid, sodium or potassium acid sulphate solutions. The current efficiencies with which persulphuric acid was formed were about the same in all three solutions. Platinum was the only successful anode material. Distillation of persulphuric acid to form hydrogen peroxide was not successfully performed; distillation of potassium persulphate, once re-crystallized, gave reasonably high yields of hydrogen peroxide. In the case of persulphuric acid or sodium persulphate solutions, extremely rigorous purification of the solution would be necessary to remove suspended catalytic impurities before proceeding to the distillation. [J. W. R.]

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The following paper contains an account of some experiments which were carried out with the object of testing the current efficiencies attainable in some of the well-known methods for the production of persulphuric acid and persulphates, and the yield of hydrogen peroxide which can be obtained by the subsequent decomposition of these bodies as so produced.

Persulphuric acid was discovered by Berthelot, and its salts first prepared by Marshall (Trans. Chem. Soc., 1891, **59,** 771). The production of persulphates has received much attention, particularly by Elbs and Schönherr (Zeit. Elektrochem., 1896, **2,** 245) and Müller and Friedberger (Zeit. Elektrochem., 1902, **8,** 230). The Consortium für Elektrochemische Industrie have patented methods for the production of persulphuric acid by electrolysis of sulphuric acid of sp. gr. 1.3–1.5 (German Patents 216538 and

[1] Manuscript received August 5, 1920.

[2] Research Chemists, Shawinigan Laboratories, Limited, Shawinigan Falls, Quebec, Canada.

217539 of 1905, British Patent 23548 of 1910). In the latter patent, hollow cooled anodes are recommended. It is claimed that the temperature of the electrolyte may even become 17°–25° with advantage, provided the anode itself be cooled down to a sufficiently low temperature.

Hydrogen peroxide is technically prepared from persulphuric acid or the persulphates by distillation with sulphuric acid under reduced pressure. Thus Elbs (Zeit. für Elektrochem., 1895, **1,** 471) showed that when persulphuric acid was distilled with sulphuric acid under reduced pressure, hydrogen peroxide resulted, but that considerable losses of oxygen took place. The causes of this loss were traced by Price and Friend (J. Chem. Soc., **85,** 1526) to the effect of traces of catalyst in the mixture of hydrogen peroxide and persulphuric acid, or Caro's acid.

In order to prevent the losses on distillation due to these catalytic impurities, Teichner (U. S. P. 916900, 1909) electrolyzes sulphuric acid to form persulphuric acid, using in the anode chamber a small auxiliary cathode, through which a small fraction of the current is allowed to pass, and which brings about the deposition of the disintegrated or dissolved platinum which would otherwise cause the hydrogen peroxide to decompose on distillation. Löwenstein (U. S. P. 1013791, 1912) describes a continuous process of distillation which rapidly separates the hydrogen peroxide from the impure solution and does not allow the incoming solution to be further contaminated by the catalyst in the spent liquor flowing out of the apparatus.

Pietzsch and Adolph (Br. Pat. 23158, 1910) eliminate the effect of impurities in a different way. Instead of using persulphuric acid as the medium, they prepare potassium persulphate electrolytically and eliminate the impurities by crystallization. The potassium persulphate is then decomposed by sulphuric acid, the $KHSO_4$ formed being filtered off from the resulting solution of hydrogen peroxide. In this way they claim to obtain a 30 percent solution with almost theoretical yield. The reactions which take place are the following:

(1.) $K_2S_2O_8 + H_2SO_4 \rightarrow K_2S_2O_7 + H_2SO_5$
(2.) $H_2SO_5 + H_2O \rightarrow H_2SO_4 + H_2O_2$
(3.) $K_2S_2O_7 + H_2O \rightarrow 2KHSO_4$

Pietzsch and Adolph (B. P. 23660, 1910) further describe a method in which the re-crystallized potassium persulphate is directly distilled in vacuo with sulphuric acid, in which case the liberated hydrogen peroxide is distilled off as fast as formed, and so does not react with persulphuric acid or Caro's acid with loss of oxygen, according to the equations

(1.) $H_2S_2O_8 + H_2O_2 \rightarrow 2H_2SO_4 + O_2$
(2.) $H_2SO_5 + H_2O_2 \rightarrow H_2SO_4 + H_2O + O_2$

Recently Levin (U. S. P. 1299485, 1919) has patented a method for the production of hydrogen peroxide through the medium of sodium persulphate obtained by electrolysis of sodium bisulphate. Sodium persulphate differs from potassium persulphate in being very much more soluble in water. In this case re-crystallization is not feasible, owing to high solubility of the sodium persulphate. The suspended impurities are gotten rid of by filtering through porous earthenware slabs, the electrolyte being periodically treated with $H_2S$ to remove dissolved platinum or lead. It is pointed out that on distillation of sodium or potassium persulphate with sulphuric acid, the hydrolysis forming hydrogen peroxide is much slower than in the case of persulphuric acid, thus allowing the hydrogen peroxide to distill off as fast as formed and largely eliminating the decomposition due to the interaction of both persulphuric acid and Caro's acid.

## PRELIMINARY EXPERIMENTS.

Attempts were first made to electrolyze sulphuric acid without the use of a diaphragm. These, however, resulted in low efficiencies, and were abandoned in favor of the use of a porous clay anode cell. Attempts were also made to get away from the necessity of using a platinum anode, the cathode in all cases consisting of a lead plate. The following substitutes were tried for the platinum anode: Gold, lead peroxide (Planté accumulator plate), lead peroxide deposited on platinum, fused $Fe_3O_4$, manganese dioxide deposited on platinum, platinum plated on copper which had first received a deposit of gold. Gold was found to be entirely too easily attacked. The lead peroxide invariably dissolved in the persulphate formed and precipitated as lead sulphate.

The $Fe_3O_4$ went readily into solution, whilst the platinum plated on gold and copper revealed the presence of minute pores, allowing attack on the gold and copper base and speedily bringing about disintegration of the deposit. The manganese dioxide was without effect on the efficiencies, and suggested the possibility of a compact electrode of this material. (See also Laurie, Chem. Trade J., 1919, **64,** 453; C. A., 1919, **13,** 1904.) As no such material was available at the time, recourse was therefore had in the following experiments to the use of a polished platinum anode.

## PREPARATION OF PERSULPHURIC ACID.

The experiments on persulphuric acid were carried out in a small cell which consisted of a porous anode compartment of about 500 c.c. capacity immersed in the electrolyte contained in a beaker, the beaker being cooled externally and the anode compartment cooled internally by means of a glass coil. The anode consisted of a platinum plate of 20 sq. cm. area, except in the case in which the anode itself was cooled, when a platinum crucible was used carrying a stopper with inlet and outlet tubes for the cooling liquid.

The following tables give typical results obtained:

### Table I.

The cell was cooled externally with ice and salt, and the anode cooling coil with circulating cold brine. The plate electrode was used. Current density 51 amp. per sq. dm. Average temp. 11° C. Anode volume 275 c.c.

| Time hr. min. | Voltage | Amperes | Total Wt. $H_2S_2O_8$ Gr. | Wt. $H_2S_2O_8$ per 100 c.c sol. gr. | Percent $H_2S_2O_8$ by wt. | Amp. Eff. Percent |
|---|---|---|---|---|---|---|
| 0: | 8.20 | 10.40 | .... | .... | .... | .... |
| 0:15 | 8.25 | 10.20 | 6.14 | 2.23 | 1.55 | 66.40 |
| 0:30 | 8.29 | 10.16 | 12.37 | 4.53 | 3.16 | 66.40 |
| 0:45 | 8.35 | 10.08 | 18.55 | 6.80 | 4.75 | 66.90 |
| 1:00 | 8.34 | 10.13 | 23.50 | 8.65 | 6.05 | 63.60 |
| 1:15 | 8.36 | 10.11 | 27.90 | 10.30 | 7.20 | 60.75 |
| 1:30 | 8.40 | 10.17 | 30.54 | 11.30 | 7.90 | 54.80 |
| 1:45 | 8.46 | 10.17 | 34.08 | 12.66 | 8.81 | 52.30 |
| 2:00 | 8.47 | 10.20 | 35.77 | 13.31 | 9.31 | 48.10 |
| 2:15 | 8.48 | 10.18 | 37.45 | 14.01 | 9.80 | 44.70 |
| 2:30 | 8.47 | 10.16 | 37.92 | 14.22 | 9.95 | 40.70 |
| 2:45 | 8.46 | 10.20 | 38.85 | 14.65 | 10.25 | 37.90 |
| 3:00 | 8.45 | 10.20 | 38.70 | 14.64 | 10.24 | 34.90 |

## Table II.

The cell was cooled in ice water, and the hollow cooled electrode used, through which ice water was circulated. The electrolyte was the same as in Table I. Current density 66 amp. per sq. dm. Average temp. 17° C. Anode volume 400 c.c.

| Time hr. min. | Av. Amp. | Av. Volts | $H_2S_2O_8$ by analysis Gr. | $H_2S_2O_8$ per 100 c.c sol. gr. | Percent $H_2S_2O_8$ by wt. | Theor. $H_2S_2O_8$ | Amp. Eff. |
|---|---|---|---|---|---|---|---|
| 0: | 6.0 | 7.2 | | | | | |
| 0:15 | 6.1 | 7.4 | 2.13 | 0.53 | 0.36 | 5.56 | 38.4 |
| 0:30 | 6.1 | 7.3 | 7.34 | 1.83 | 1.29 | 11.19 | 61.6 |
| 0:45 | 6.05 | 7.2 | 10.88 | 2.71 | 1.91 | 16.68 | 64.9 |
| 1:00 | 6.0 | 7.2 | 13.67 | 3.44 | 2.42 | 22.05 | 62.1 |
| 1:15 | 6.0 | 7.1 | 17.15 | 4.24 | 2.99 | 27.34 | 62.8 |
| 1:30 | 6.0 | 7.1 | 20.08 | 5.08 | 3.58 | 32.80 | 61.4 |
| 1:45 | 6.0 | 7.1 | 22.32 | 5.65 | 3.99 | 37.28 | 59.8 |
| 2:00 | 6.0 | 7.1 | 24.35 | 6.20 | 4.25 | 43.75 | 55.7 |
| 2:15 | 6.0 | 7.1 | 26.40 | 6.74 | 4.75 | 49.21 | 53.6 |
| 2:30 | 6.0 | 7.1 | 27.65 | 7.08 | 5.00 | 54 67 | 50.8 |
| 3:00 | 6.0 | 7.05 | 29.55 | 7.58 | 5.35 | 65.81 | 45.0 |
| 3:15 | 6.0 | 7.05 | 30.80 | 7.95 | 5.60 | 71.09 | 42.3 |
| 3:30 | 6.0 | 7.0 | 31.50 | 8.15 | 5.75 | 76.55 | 41.2 |

## Table III.

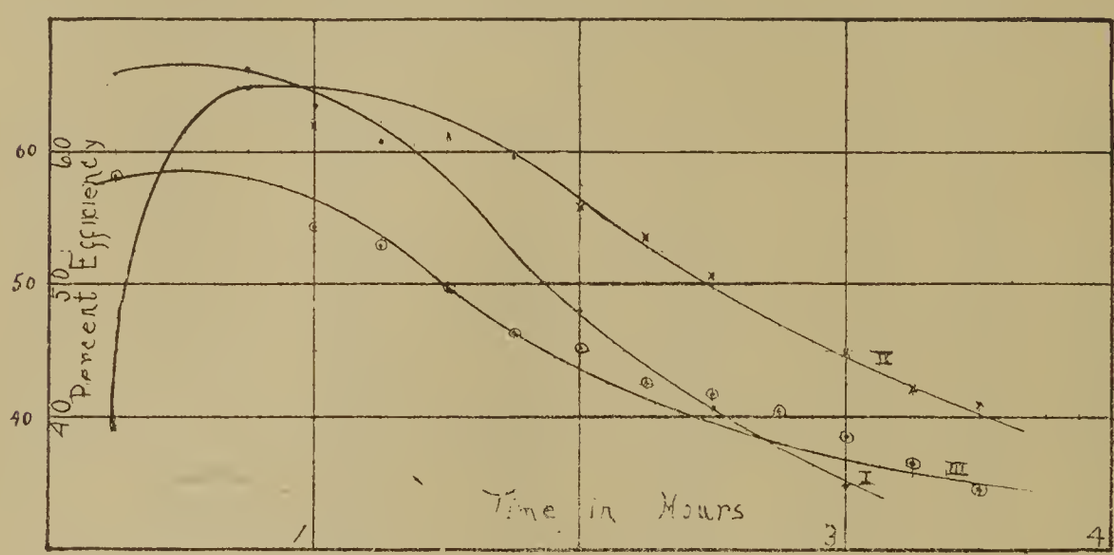
The cell and hollow cooled electrode were cooled in tap water which was at 22° C. Average temp. 28° C. Current density 62 amp. per sq. dm. Anode volume 400 c.c.

| Time hr. min. | Av. Amp. | Av. Volts | $H_2S_2O_8$ by analysis Gr. | $H_2S_2O_8$ per 100 c.c sol. gr. | Percent $H_2S_2O_8$ by wt. | Theor. $H_2S_2O_8$ | Amp. Eff. |
|---|---|---|---|---|---|---|---|
| 0:15 | 6.2 | 6.2 | 3.29 | 0.63 | 0.59 | 5.65 | 58.2 |
| 1:00 | 6.1 | 6.1 | 12.05 | 3.06 | 2.20 | 22.20 | 54.4 |
| 1:15 | 6.1 | 6.06 | 14.69 | 3.74 | 2.64 | 27.75 | 53.0 |
| 1:30 | 6.1 | 6.06 | 16.55 | 4.23 | 2.98 | 33.35 | 49.7 |
| 1:45 | 6.1 | 6.05 | 18.03 | 4.62 | 3.26 | 38.90 | 46.4 |
| 2:00 | 6.06 | 6.04 | 20.25 | 5.20 | 3.66 | 44.20 | 45.4 |
| 2:15 | 6.06 | 6.04 | 21.15 | 5.45 | 3.84 | 49.70 | 42.6 |
| 2:30 | 6.06 | 6.04 | 22.98 | 5.93 | 4.18 | 55.20 | 41.7 |
| 2:45 | 6.05 | 6.06 | 24.42 | 6.30 | 4.44 | 60.07 | 40.4 |
| 3:00 | 6.08 | 6.07 | 25.67 | 6.65 | 4.60 | 66.50 | 38.6 |
| 3:15 | 6.07 | 6.09 | 26.35 | 6.85 | 4.83 | 72.00 | 36.6 |
| 3:30 | 6.07 | 6.10 | 26.94 | 7.00 | 4.92 | 77.50 | 34.77 |

Comparison of these tables shows that with hollow cooled electrodes almost as good results may be obtained with an electrolyte temperature rising to 30° C. as with a temperature of 12° C. when the electrode is not cooled.

The volume of anolyte was smaller in Table I than in Tables II and III; hence the higher concentration reached in that experiment.

The results of these tests are shown graphically in the accompanying figure.




DISTILLATION OF PERSULPHURIC ACID.

The persulphuric acid obtained in accordance with the methods outlined was filtered through paper and distilled in vacuo in an all-glass apparatus. It was found, however, that practically no hydrogen peroxide was obtained, and as at the time, due to war exigencies, no adequate filtering media were available, the distillation of persulphuric acid was abandoned, and attention turned to the potassium salts.

PREPARATION OF POTASSIUM PERSULPHATE.

In the first experiments on the preparation of potassium persulphate the cell used was similar to that used in the experiments on persulphuric acid, with the exception that a saturated solution of $KHSO_4$ was used as electrolyte. It was found that the impoverishment of the solution due to precipitation of $K_2S_2O_8$ led to very low current efficiencies. In order to overcome this, a continuous type cell was constructed. The electrolyte was contained in a well-cooled reservoir, from which it flowed through the anode compartment of the cell, and, together with the precipitated potas-

sium persulphate, flowed away from the bottom of the anode compartment to a filter which removed the potassium persulphate, the filtered electrolyte being re-saturated with potassium bisulphate and returned to the reservoir. The anode compartment was cooled by means of a coil, and the whole cell jacketed with an ice-salt cooling mixture. The hollow cooled anode was also tried, but did not give results as good as those obtained by the use of a platinum plate as anode. The cell was allowed to run until the whole of the solution employed became saturated with potassium persulphate.

The following results obtained in a 6-hour continuous run are typical:

TABLE IV.

Average temperature, anode compartment, 13° C. Average rate of circulation, 3.8 liters per hour. Volume of liquor in circulation, 3 liters, saturated at the temperature of the reservoir. Volume of anode compartment, 300 c.c.

| Time hr. | Av. Amp. | Av. Volts | Dry Wt. of ppt. g. | Percent $K_2S_2O_8$ in Sample | Actual Weight $K_2S_2O_8$ Percent | Percent Eff. per each hr. | Amp. Eff. for Run |
|---|---|---|---|---|---|---|---|
| 1 | 10.0 | 8.1 | 33.4 | 76.6 | 25.6 | 50.8 | 50.8 |
| 2 | 9.9 | 9.6 | 36.2 | 63.6 | 23.0 | 46.2 | 49.0 |
| 3 | 10.1 | 10.1 | 34.0 | 85.0 | 28.6 | 56.2 | 51.5 |
| 4 | 9.9 | 10.9 | 31.6 | 77.0 | 24.4 | 49.0 | 50.9 |
| 5 | 9.9 | 11.2 | 26.0 | 77.5 | 20.2 | 40.5 | 48.7 |
| 6 | 10.1 | 14.6 | 26.9 | 82.6 | 22.2 | 44.5 | 48.0 |

In one run 1 g. of KCN was added per liter of electrolyte, as this substance is one of those which have been recommended as a useful depolarizer. It was found, however, in this case to be without any marked beneficial effect on the yield.

The potassium persulphate obtained varied a good deal in crystal size; the finer precipitates obtained were filtered only with difficulty, and tenaciously retained mother liquor. This explains the considerable differences in the percentage composition of the dried precipitate obtained, the balance of the samples consisting of $KHSO_4$. On one recrystallization of the crude material a product analyzing 99.9 percent $K_2S_2O_8$ was obtained.

## DISTILLATION OF POTASSIUM PERSULPHATE.

The potassium persulphate was mixed with sulphuric acid and water, and distilled from a glass distilling flask, which was arranged so that the vapors could not come in contact with rubber. The distillate was collected in a well-cooled receiver, which in turn was in communication with a good pump and manometer.

The first experiment on this distillation was carried out on a sample of Kahlbaum C. P. material analyzing 99.35 percent $K_2S_2O_8$. When the procedure had been modified to obtain the best results with this material, distillations were made on the once-recrystallized potassium persulphate made by the procedure before outlined, and also on the crude uncrystallized material. In the case of the Kahlbaum material and of the recrystallized material, 100 g. of persulphate was mixed with 67 c.c. of 1.6 sp. gr. $H_2SO_4$ and 50 c.c. of water, these proportions being those quoted by Pietzsch and Adolph (*loc. cit.*). As the distillation proceeded, water was continuously added so as to keep the total volume constant in the distilling flask. In the case of the uncrystallized material, an amount was taken which contained 100 g. of $K_2S_2O_8$, and a proportionately larger amount of water was added at the start. The distillate was collected in two separate receivers, the first half giving a fairly concentrated hydrogen peroxide solution, the second half being a weaker solution. The runs were continued till the residue showed no potassium persulphate. The following table contains the results obtained:

TABLE V.

| Material | Vol. distillate, 1. c.c. | Percent $H_2O_2$ distillate 1, by. wt | Volume distillate 2. c.c. | Percent $H_2O_2$ distillate 2 | Total vol. distillate. c.c. | Total wt. $H_2O_2$ | Yield. Percent |
|---|---|---|---|---|---|---|---|
| Kahlbaum ..... | 41 | 13.17 | 86 | 3.59 | 127 | 10.3 | 81.7 |
| Recryst. ..... | 95 | 9.31 | 72 | 1.04 | 167 | 9.9 | 78.4 |
| 71.6% crude ..... | 140 | 3.42 | 15 | 0.18 | 155 | 4.8 | 38.3 |
| 80.8% " ..... | 136 | .... | .. | ... | 136 | 6.2 | 48.9 |

In some cases the material in the distilling flask showed considerable tendency to foam.

### PREPARATION OF SODIUM PERSULPHATE.

This was effected in a cell having a porous porcelain anode compartment of 600 c.c. capacity, provided with cooling coils through which brine was circulated. The electrolyte consisted of a solution of $NaHSO_4$ saturated at the temperature of the cell. It was found that improved efficiencies were obtained by agitation of the liquor around the anode by means of an air current. The current efficiencies obtainable were better than with the potassium salt previously referred to, and although in the formation of persulphuric acid the current efficiencies were somewhat higher in the early stages of a run, the efficiencies in the case of the sodium salt appeared to be much better maintained than in the case of persulphuric acid.

The following table gives a typical run:

TABLE VI.

Current density 65 amp. per sq. dm. Average temp, 11° C. Anode volume 600 c.c.

| Time | Av. Amp. | Av. Volts | $Na_2S_2O_8$ per 100 c.c. | $Na_2S_2O_8$ total. g. | Theoretical wt. | Yield, percent |
|---|---|---|---|---|---|---|
| 1 | 13.0 | 10.3 | 5.53 | 33.15 | 57.7 | 57.5 |
| 2 | 13.8 | 9.6 | 11.07 | 66.40 | 119.0 | 55.7 |
| 3 | 13.6 | 9.7 | 16.65 | 99.60 | 179.4 | 55.5 |
| 4 | 13.2 | 10.6 | 19.55 | 117.00 | 237.8 | 49.2 |
| 5 | 12.6 | 10.9 | 23.40 | 140.00 | 293.8 | 47.8 |
| 6 | 12.5 | 11.0 | 27.4 | 164.3 | 349.3 | 47.0 |

It will be noticed that the concentration of sodium persulphate rose as high as 27 percent in about six hours. In one case, however, it was impossible to get a concentration above about 9 percent $Na_2S_2O_8$, the persulphate crystallizing out as formed. In this case 230 g. salt crystallized out containing over 53.4 percent $Na_2S_2O_8$. It is possible that some double salt was here precipitated.

### DISTILLATION OF SODIUM PERSULPHATE.

The apparatus used for this distillation was the same as that described under the distillation of potassium persulphate.

The material used was the concentrated solution of sodium

persulphate obtained from the electrolytic apparatus, and contained in all cases above 25 g. $Na_2S_2O_8$ per 100 c.c. The solution from the electrolyser was filtered through a fine grade alundum filtering cone to remove catalytic impurities. The ratio of acidity to persulphate was varied over a wide range, and the amount of water present was also varied. In no case, however, could a yield of hydrogen peroxide be obtained above 9 percent of that theoretically possible from the sodium persulphate taken. The following table shows the materials distilled and the yields obtained:

TABLE VII.

| Volume $Na_2S_2O_8$ taken. c.c. | Weight $Na_2S_2O_8$ g. | $H_2SO_4$ added. g. | Water added c.c. | Total acid in g. $H_2SO_4$ | Yield $H_2O_2$. Percent |
|---|---|---|---|---|---|
| 250 | 69.5 | 0 | 0 | 38 | 4.3 |
| 220 | 60.5 | 0 | 0 | 33.5 | 4.3 |
| 250 | 68.4 | 90 | 0 | 128 | 4.5 |
| 155 | 39.2 | 377 | 0 | 400 | 7.9 |
| 154 | 48.8 | 340 | 50 | 400 | 9.0 |

Some attempts were made to improve the above yields by first heating the filtered solution of sodium persulphate under pressure in a bomb. The results showed that on heating in a bomb, amounts of hydrogen peroxide up to 23 percent of the theoretical were generated, but on subsequent distillation of these solutions the yield of hydrogen peroxide fell to below 7 percent, and are thus in the same class as the direct distillations already quoted.

It is clear that filtration as a means of removing catalytic impurities would need to be effected through an extremely carefully graded filtering medium in order to attain purification comparable to a single recrystallization of the potassium salt.

## SUMMARY.

1. Of the materials experimented with as anode material, platinum was the only one offering possibilities of success, with the possible exception of manganese dioxide.

2. The current efficiencies in the production of persulphuric acid, potassium persulphate and sodium persulphate show no great advantage for any one of them over the others.

3. Sodium persulphate solutions can apparently be produced to a much higher concentration than can persulphuric acid, without sacrifice of current efficiency. The potassium salt is, of course, obtained in solid form.

4. Distillation of once recrystallized potassium persulphate with sulphuric acid and water gives reasonably high yields of hydrogen peroxide.

5. Distillation of persulphuric acid or sodium persulphate solutions would need to be preceded by extremely rigorous purification to remove catalytic impurities.

*Shawinigan Laboratories, Limited,*
*Shawinigan Falls, Quebec, Canada,*
*August 3, 1920.*

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## DISCUSSION.

H. C. Cooper[1]: I was sorry to be informed the other day that this work is being discontinued; because, for the sake of the advance of American electrochemistry, it seems as though such a work ought to go on even in the face of difficulties. One who has read German literature is impressed with the large number of patents taken out along this line. We do not know and cannot say, at this distance, how successful these are. They may be purely academic, as has been intimated with reference to certain other remarks today, but apparently the Germans are going ahead with this method. The difficulty seems to be getting rid of the fine platinum. I recall three methods that they resorted to; one is treating the platinum beforehand, making it as resistive as possible, while the others involve the removal of the fine platinum. The removal was accomplished either by electrolytic methods, which they claim are very satisfactory, or by the introduction of a rod of aluminum. I am sorry Dr. Skirrow is not here, or some of his associates, that we might ask them if they tried these meth-

[1] Asst. Prof. of Chemistry, College City of New York. New York City.

ods. I should like to see all possible means exhausted before we give up an electrolytic process.

F. W. SKIRROW[2] *(Communicated)*: The reason that the work was temporarily discontinued was not on account of the technical difficulties alluded to in the paper, which could be readily minimized or overcome, but on account of the present somewhat unfavorable outlook for the method from an economic standpoint.

[2] Chief Chemist, Shawinigan Laboratories, Ltd., Shawinigan Falls, Que.

*A paper presented at the Thirty-eighth General Meeting of the American Electrochemical Society, in Cleveland, October 2, 1920, President Landis in the Chair.*

# THE INFLUENCE OF COPPER, MANGANESE AND CHROMIUM AND SOME OF THEIR COMBINATIONS ON THE CORROSION OF IRON AND STEEL[1]

By E. A. Richardson[2] and L. T. Richardson.[3]

During the past several years a considerable amount of work has been done upon the corrosion of copper-bearing steels and copper-bearing irons, but the ferrous base of the alloys made has been normal steel or iron to which different amounts of copper were added. As a result, copper has been the only variable investigated, and hence only the effect of copper has been determined. The most comprehensive work has been done by Mr. D. M. Buck,[4] who has added various amounts of copper to a low carbon steel containing about 0.45 percent of manganese, and his results indicate that, as far as corrosion is concerned, little benefit results from adding more than about 0.25 percent of copper to such a steel. Buck finds that sulphur is very injurious to iron or steel from a rust-resisting standpoint, and believes that copper retards corrosion by neutralizing the harmful effects of sulphur.

A. S. Cushman,[5] on the other hand, contends that copper has no beneficial effect when added to a commercially pure iron, while Burgess and Aston[6] and Kalmus and Blake[7] find that copper is beneficial to pure iron.

The authors of most of the text books on iron and steel consider manganese as detrimental to steel as regards corrosion resistance, but many of the authorities on corrosion are at the present time not so certain as to this. Cushman, since his work on the corrosion of fence wire, has always contended that manganese is harmful to iron, and, as a result, has been a strong advocate of an iron as low as possible in this element. However, Cushman

[1] Manuscript received August 6, 1920.

[2] National Lamp Works, General Electric Co., Cleveland, Ohio.

[3] Chemical Engineer, New Brunswick, N. J.

failed to analyze his samples of fence wire for copper, which is unfortunate, since O. W. Storey[8] later found that certain specimens of fence wire that had shown great resistance to corrosion contained copper, and reached the additional conclusion that, as far as corrosion is concerned, manganese is neutral. In view of what is now known as to the effects of copper in steel on atmospheric corrosion, it is very probable that Cushman's conclusions as to the rapid corrosion of fence wire are somewhat at fault. However, this does not detract from the value of his paper, since it was written some years ago, before the corrosion of iron was being intensively investigated, and was, no doubt, of great value in calling attention to the importance of the corrosion problem and of stimulating work upon it.

Chromium has usually been considered as beneficial to the rust-resisting properties of iron, although little work has been done.

In some of our previous work[9] we noticed that copper makes iron red-short, and that the fracture has every appearance of being an inter-grained one. Further, this red-shortness is eliminated by the addition of manganese or chromium, the amount necessary depending upon the amount of copper present. It was therefore thought that manganese or chromium might likewise exert some influence in increasing the effect that copper has of decreasing corrosion as it does the physical property of forging.

In order to investigate this, tests were made upon steel, both free from and containing copper, and upon commercially pure iron, both free from and containing copper. These tests brought out a number of interesting deductions, as follows: Steel rusts much faster than iron—which fact has been long known. The presence of copper, however, decreases the corrosion of both iron and steel, but its action is more intense and pronounced in the case of the latter, so that for a given amount of copper the steel is actually more resistant to atmospheric corrosion than is the corresponding copper-bearing iron. This is true, even though the reverse is true for the copper-free materials. It was, moreover, concluded that it was the manganese present in the steel, but not in the iron, that was intensifying the action of the copper in reducing corrosion. It was also pointed out that the observations of Cushman that copper had no effect on the corrosion of pure iron, and of Buck that it exerted a marked retarding influence on the

TABLE I.

*Corrosion of Copper Manganese Alloys of Iron.*

| Alloy | Percent | | Loss in 8 Months gm./sq.cm. | Character of Metal Surface after Cleaning. 8 Months' Exposure. | Character of Rust Coating After Two and One-half Years | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | Texture | | | | | Color | | | Appearance | |
| | Cu | Mn | | | Very Smooth | Smooth | Med. | Coarse | Very Coarse | Light | Med. | Dark | Tight | Loose |
| A | 0.04 | 0.00 | 0.03960 | Coarse deep pits..... | ..... | ..... | ..... | ..... | x | x | ..... | ..... | ..... | x |
| F | 0.04 | 0.44 | 0.03672 | Coarse deep pits..... | ..... | ..... | ..... | ..... | x | x | ..... | ..... | ..... | x |
| K | 0.04 | 0.93 | 0.03770 | Coarse deep pits..... | ..... | ..... | ..... | x | ..... | ..... | x | ..... | ..... | x |
| G | 0.04 | 1.59 | 0.03650 | Large pits but not as deep.. | ..... | ..... | ..... | ..... | x | x | ..... | ..... | ..... | x |
| L | 0.04 | 1.75 | 0.03510 | Large pits but not as deep.. | ..... | ..... | ..... | ..... | x | x | ..... | ..... | ..... | x |
| J | 0.05 | 3.11 | 0.02528 | Fine pits ..... | ..... | ..... | x | ..... | ..... | ..... | x | ..... | x | x |
| Q | 0.04 | 3.00 | 0.02550 | Fine pits ..... | ..... | ..... | x | ..... | ..... | ..... | x | ..... | x | x |
| Aver. | 0.04 | | | | | | | | | | | | | |
| F1 | 0.36 | 0.69 | 0 03015 | Pits large but shallow.... | ..... | ..... | x | ..... | ..... | ..... | x | ..... | x | x |
| E1 | 0.30 | 0.96 | 0.02840 | Pits large but shallow.... | ..... | x | x | ..... | ..... | ..... | ..... | x | x | x |
| C1 | 0.36 | 1.13 | 0.02868 | Pits large but shallow.... | ..... | x | x | ..... | ..... | ..... | ..... | x | x | x |
| S1 | 0.32 | 2.34 | 0.02698 | Pits small and shallow.... | ..... | x | x | ..... | ..... | ..... | ..... | x | x | x |
| Aver. | 0.33 | | | | | | | | | | | | | |
| P | 0.48 | 1.32 | 0.02928 | Medium-sized deep pits.... | ..... | ..... | x | ..... | ..... | ..... | x | ..... | x | x |
| R | 0.50 | 1.29 | 0.02660 | Medium-sized shallow pits. | ..... | ..... | x | ..... | ..... | ..... | x | ..... | x | x |
| Aver. | 0.49 | | | | | | | | | | | | | |
| N | 0.92 | 0.32 | 0.02505 | Large shallow pits..... | ..... | ..... | x | ..... | ..... | ..... | x | ..... | x | x |
| H1 | 1.00 | 1.81 | 0.02445 | Few shallow pits..... | x | x | ..... | ..... | ..... | ..... | ..... | x | x | ..... |
| Aver. | 0.96 | | | | | | | | | | | | | |
| H | 1.97 | 0.02 | 0.02512 | Large shallow pits..... | ..... | x | x | ..... | ..... | ..... | x | ..... | x | x |
| Q | 1.87 | 1.27 | 0.02345 | Few shallow pits..... | ..... | x | ..... | ..... | ..... | ..... | ..... | x | x | ..... |
| Aver. | 1.92 | | | | | | | | | | | | | |
| T | 3.22 | 0.06 | 0.02658 | Medium deep pits..... | ..... | ..... | x | ..... | ..... | ..... | x | ..... | x | ..... |
| S | 3 03 | 2.78 | 0.02130 | Very few small shallow pits | ..... | x | ..... | ..... | ..... | ..... | ..... | x | x | ..... |
| Aver. | 3.12 | | | | | | | | | | | | | |
| M | 4.65 | 0.04 | 0.02600 | Medium shallow pits..... | ..... | ..... | x | ..... | ..... | ..... | x | ..... | x | x |
| Z | 0.62 | 0.48 | 0.02845 | Large shallow pits..... | ..... | x | x | ..... | ..... | ..... | x | ..... | x | x |
| E | 1.63 | 1.13 | 0.02465 | Small shallow pits..... | ..... | x | x | ..... | ..... | ..... | x | x | x | x |
| W | 3.54 | 2.51 | 0.02117 | Surface smooth ..... | ..... | x | ..... | ..... | ..... | ..... | ..... | x | x | ..... |

corrosion of steel, might be reconciled by this reasoning. More recently the results of the official test of the American Society for Testing Materials[10] show similar facts.

In order to investigate this idea further, a series of alloys was made by melting commercially pure iron in an electric furnace and adding the necessary amounts of the carbon-free alloying elements so as to produce a product low in carbon. The analyses of these alloys are given in Table I.

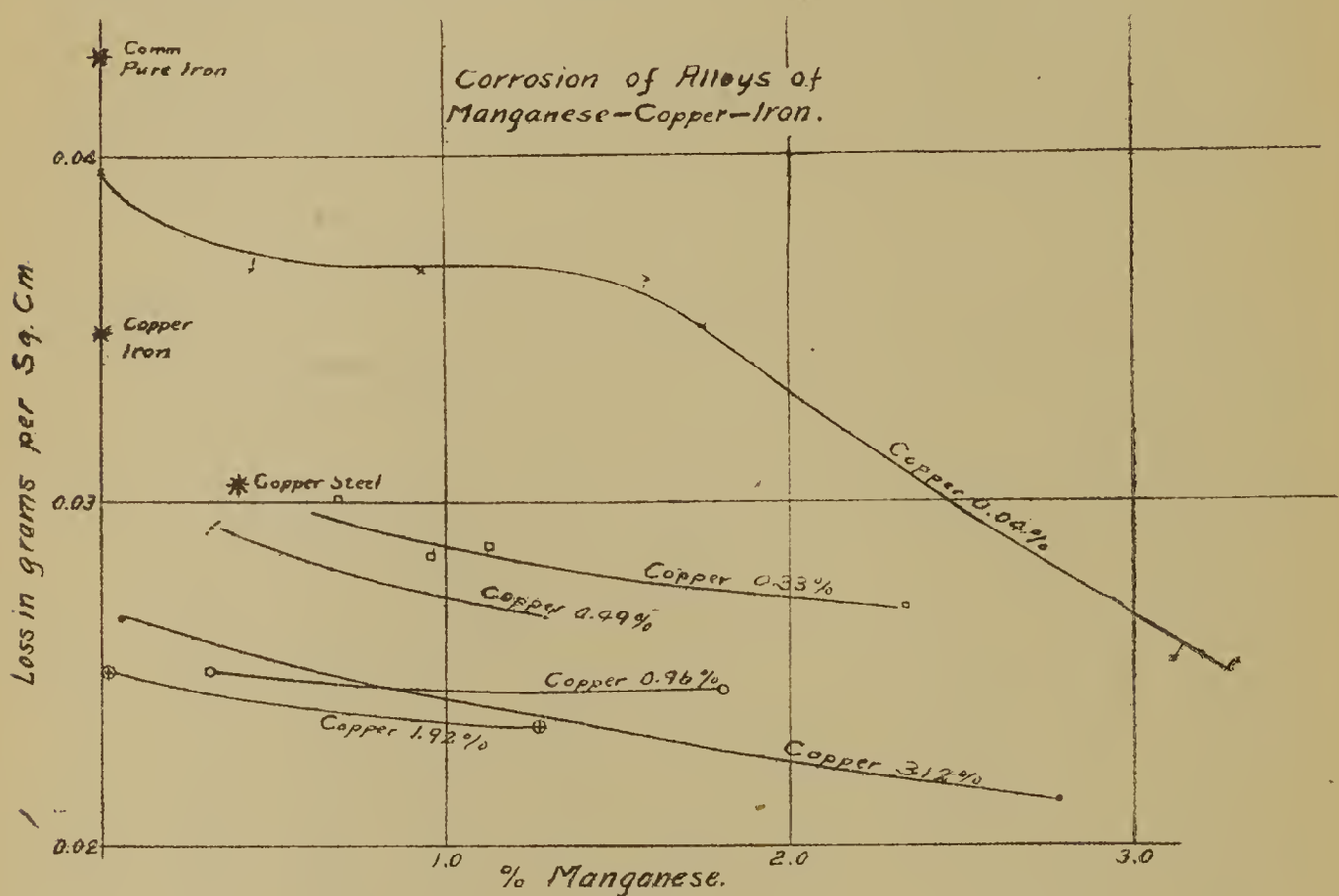



Fig. 1.

The alloys were first tested for physical properties, including those of forging. The results of these tests will be given in another paper. Briefly, the forging tests confirmed our earlier observations that copper caused iron to be red-short, and that this undesirable property could be eliminated by the addition of manganese or chromium in amounts depending on the copper content. They also showed that, up to at least 3.5 percent of copper, this element does not affect the cold-working properties, but that the addition of manganese causes hardness, until with more than about 2 percent the alloys are too brittle to work, and that with more than 2 percent of chromium the alloys are hard and tough, but not brittle.

After the ingots were forged they were cut into test pieces about 1 in. x 2 in. x ⅛ in. (2.5 x 5 x 0.3 cm.) thick for corrosion tests. These pieces were thoroughly annealed at 750° C., were pickled in acid to remove the adhering oxide, the surface removed with a file, and finally made smooth with emery cloth. Duplicate test pieces were exposed to the atmosphere from December 6, 1916, to July 16, 1917. During the test the specimens were examined at frequent intervals for texture and color of the rust. From the very start it was evident that the alloys containing copper were superior to those that were free from copper, which showed itself in the color of the rust. This color distinction was a dark red in the case of the copper-bearing alloys, and a light red or a yellowish red in the case of the alloys in which copper was absent or present in only small amounts. In fact, it seems to be a general rule that the darker the color of the rust, the more rust-resisting the material. This connection between a dark red rust and a rust-resisting iron has been noted by other observers. The same superiority was also apparent from the texture and appearance of the rust coating. The alloys containing copper possessed a fine-grained, adherent coating, while those in which copper was absent or present in very small amounts had a coating that was coarse and non-adherent. In the alloys tested, the condition of the adhering rust varied widely, from a very loose and coarsely granular coating on a Bessemer steel to a black, almost enamel-like coating on some of the alloys of greatest rust resistance.

As before stated, at the end of eight months the specimens were taken down and the rust removed by mechanically removing as much as possible with a brush and then dissolving the remainder with a hot ammoniacal solution of ammonium citrate. After thorough drying with alcohol they were weighed, and the loss in weight was taken as an index of corrosion. The losses in weight were calculated in grams per square centimeter, and are given in Table I.

The loss in weight, however, does not always give a true index of the extent of destruction by corrosion, since the material could fail by "pitting" or localized corrosion. Hence, in order to take this factor into account, the test pieces were examined as to appearance after cleaning. These data are also included in Table I.

After weighing, one set of test pieces was exposed to the atmosphere on August 8, 1917, in the same location, and after two and one-half years the visual characteristics of the rust coating were as given in Table I. The appearance at this time was identical with that at the shorter period of eight months. At the last observation, after about three years' exposure, some of the best alloys (U1, Y1, R1) have such a closely adhering and thin rust coating that the original file marks are still visible. This method of judging the degree of corrosion is that adopted by the American Society for Testing Materials, in which the condition of the rust coating is described by its color, texture and general appearance. In this way the character of the rust can be quite accurately defined.

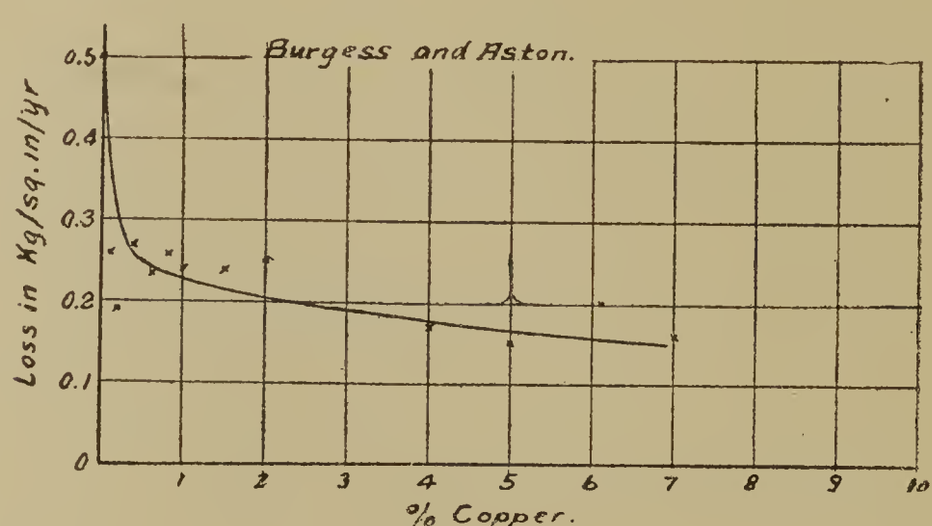



FIG. 2.

RESULTS.

*Copper Manganese Alloys.*—The specimens of these alloys may, roughly, be divided into six groups, namely, those containing 0.04, 0.33, 0.49, 0.96, 1.92 and 3.12 percent of copper. In each group the manganese varies, while the copper remains constant. The data are shown graphically in curve form in Figure 1. It is evident from the curves that each successive addition of copper lowers the amount of corrosion. The greatest decrease, however, comes with the first addition of copper. Thus an increase of copper from 0.04 to 0.33 percent gives as great an increase in corrosion resistance as does an increase from 0.33 to 1.92 percent. In addition to this, very little, if any, increase in rust resistance occurs after the copper content has passed 2 percent. This is

in line with what has been observed by Mr. D. M. Buck, who found that with the addition of successively increasing amounts of copper to steel, the greatest decrease in corrosion took place with the first additions of copper. Mr. Buck, however, worked with normal steels in which the manganese remained constant. This characteristic of the effect of copper may have some value, since it enables the property of corrosion to be made quite uniform. If very small amounts of copper are added, then slight changes from this amount will cause relatively large changes in the degree of corrosion, while if larger amounts of copper are added, small changes in the copper content will cause only small changes in the tendency to corrode.

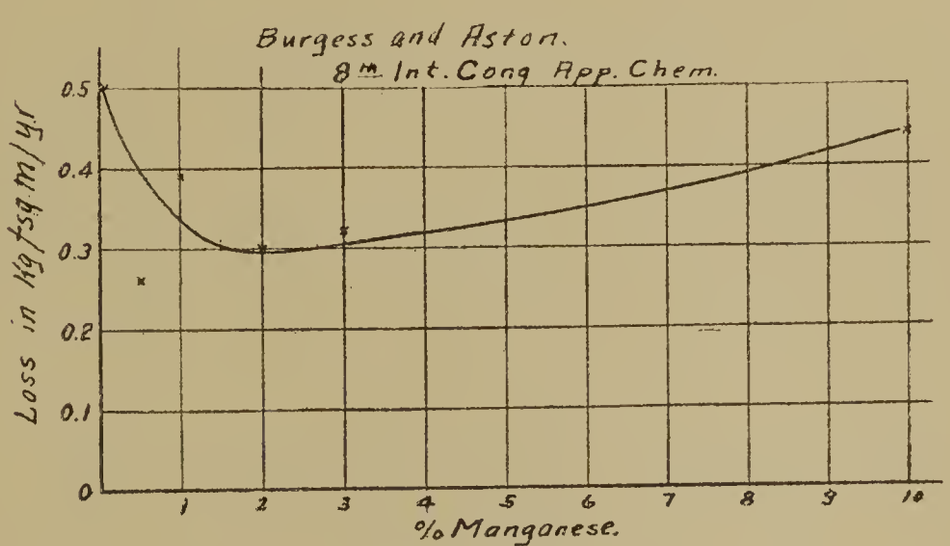



FIG. 3.

The curves also show that additions of copper to pure iron decreases the amount of corrosion. This is in line with the work of Burgess and Aston, who worked with electrolytic iron as a base. The results of their investigation are given in Figure 2. Kalmus and Blake, using commercially pure iron as a base, also found that additions of copper made this iron more rust-resisting.

Increasing the manganese content also decreases the amount of corrosion, but to a lesser extent than the copper. This is at variance with the conclusions reached by many of the past observers, who claim that manganese increases the tendency to rust. More recently, however, the idea is becoming more general that manganese is not detrimental to the rust-resisting properties of steel, but that sulphur or manganese sulphide is the harmful im-

purity. For instance, O. W. Storey, from an investigation of fence wire, reached the conclusion that, as far as corrosion is concerned, manganese is neutral. Burgess and Aston, working with manganese alloys of electrolytic iron, showed that manganese, at least, is not harmful. Their data, however, were rather erratic, and are given in Figure 3.

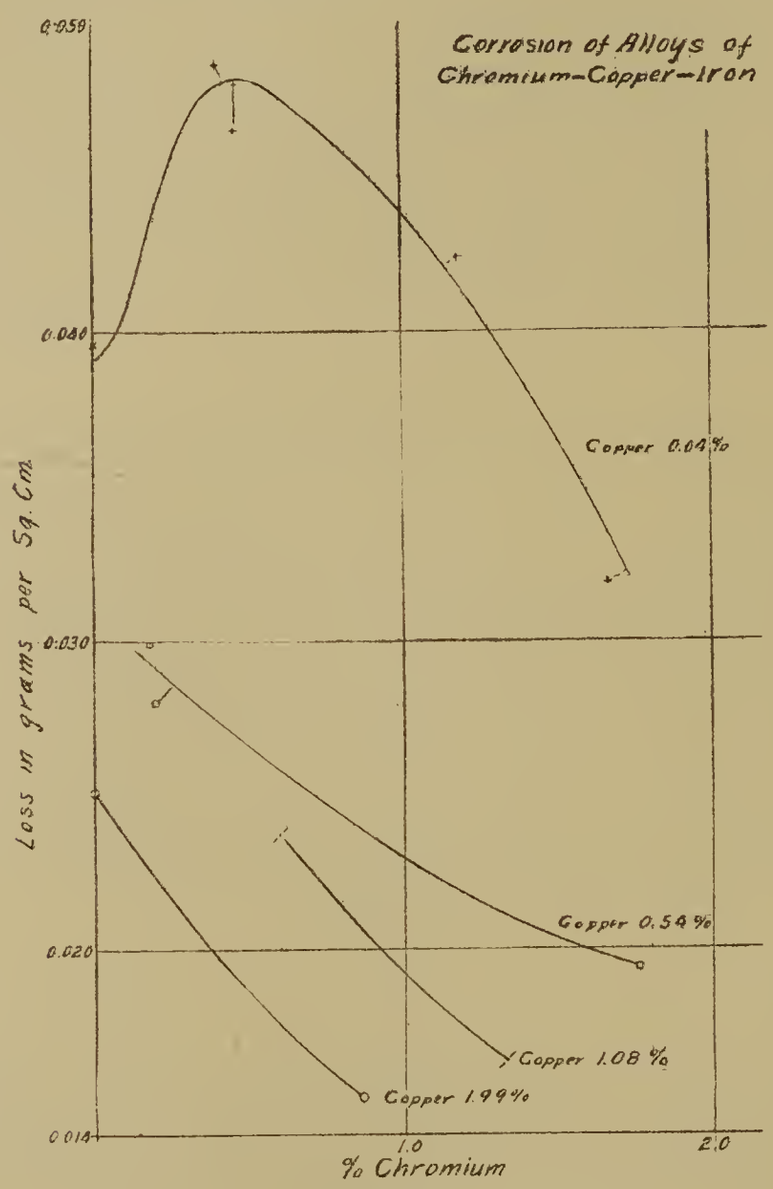



FIG. 4.

The advocates of pure iron as a rust-resisting material have always made manganese as low as possible, thinking it to be harmful. However, commercially pure iron contains about 0.04 percent of copper, which causes it to be red-short. The addition of a small amount of manganese would make this iron slightly more rust-resisting, and at the same time remove the tendency to be red-short, while the addition of still more copper and manganese would materially increase its rust-resisting properties.

## Table II.

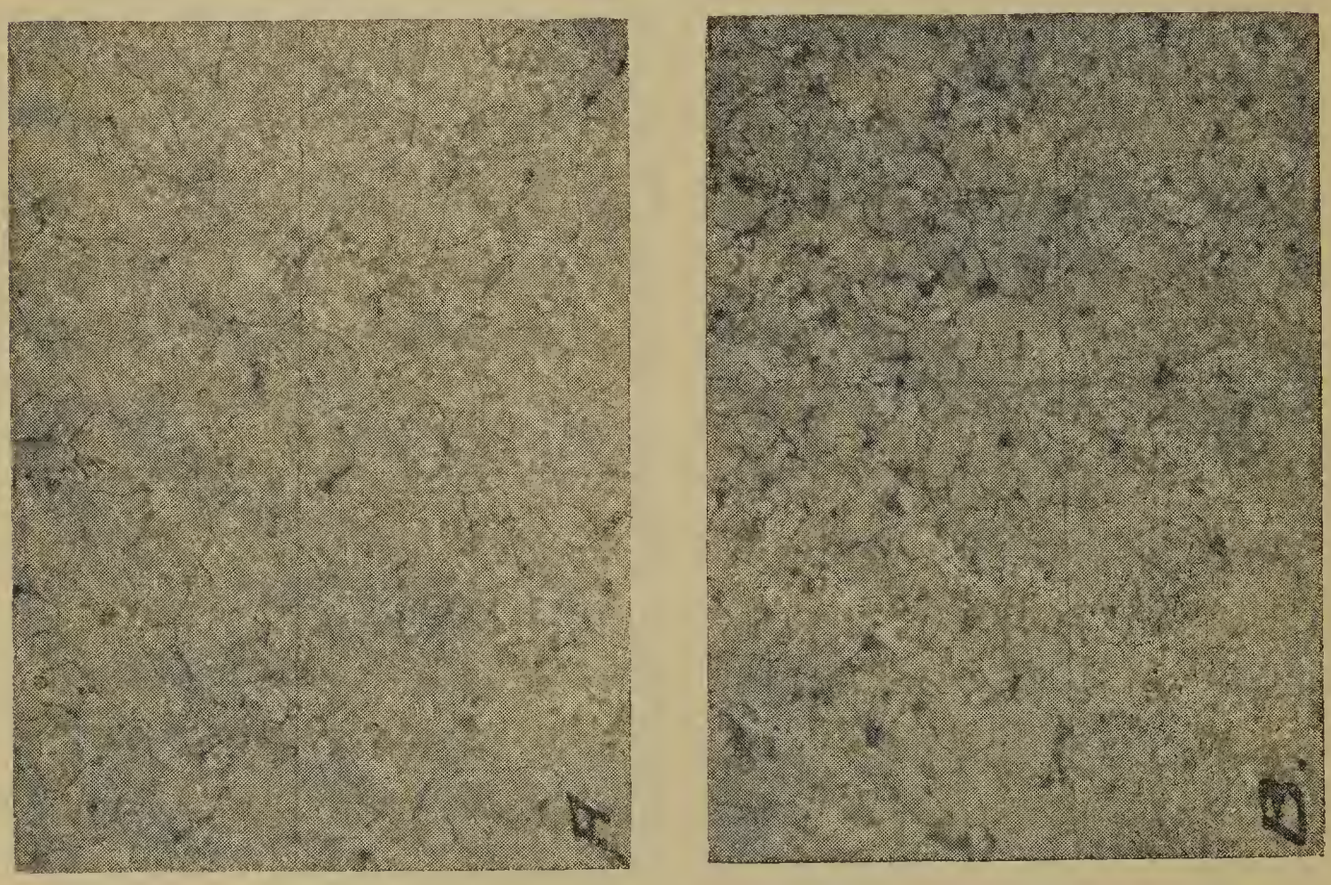
*Corrosion of Copper Chromium Alloys of Iron.*

| Alloy | Percent | | Loss in 8 Months gm./sq.cm. | Character of Metal Surface after Cleaning. 8 Months' Exposure | Character of Rust Coating After Two and One-half Years | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | Texture | | | | | Color | | | Appearance | |
| | Cu | Cr | | | Very Smooth | Smooth | Med. | Coarse | Very Coarse | Light | Med. | Dark | Tight | Loose |
| A | 0.04 | 0.00 | 0.03960 | Coarse deep pits.... | ..... | ..... | ..... | ..... | x | x | ..... | ..... | ..... | x |
| D1 | 0.04 | 0.21 | 0.04051 | Coarse deep pits.... | ..... | ..... | ..... | ..... | x | x | ..... | ..... | ..... | x |
| I1 | 0.05 | 0.40 | 0.04870 | Coarse deep pits.... | ..... | ..... | ..... | ..... | x | x | x | ..... | ..... | x |
| A1 | 0.05 | 0.46 | 0.04650 | Coarse deep pits.... | ..... | ..... | ..... | ..... | x | x | ..... | ..... | ..... | x |
| B1 | 0.04 | 1.18 | 0.04240 | Coarse shallow pits.. | ..... | ..... | ..... | ..... | x | x | ..... | ..... | ..... | x |
| N1 | 0.04 | 1.67 | 0.03190 | Large shallow pits... | ..... | ..... | ..... | x | ..... | x | ..... | ..... | ..... | x |
| Aver. | 0.04 | | | | | | | | | | | | | |
| M1 | 0.45 | 0.18 | 0.02990 | Large shallow pits... | ..... | ..... | x | ..... | ..... | ..... | x | ..... | x | x |
| L1 | 0.59 | 0.20 | 0.02800 | Large shallow pits... | ..... | ..... | x | ..... | ..... | ..... | x | ..... | x | x |
| U1 | 0.58 | 1.76 | 0.01935 | Surface quite smooth. | x | ..... | ..... | ..... | ..... | ..... | x | ..... | x | x |
| Aver. | 0.54 | | | | | | | | | | | | | |
| P1 | 1.14 | 0.60 | 0.02370 | Large shallow pits... | ..... | x | ..... | ..... | ..... | ..... | ..... | x | x | ..... |
| Y1 | 1.02 | 1.33 | 0.01635 | Few shallow pits.... | ..... | x | ..... | ..... | ..... | ..... | ..... | x | x | ..... |
| Aver. | 1.08 | | | | | | | | | | | | | |
| H | 1.97 | 0.00 | 0.02512 | Few shallow pits.... | ..... | x | ..... | ..... | ..... | ..... | ..... | x | x | ..... |
| R1 | 2.01 | 0.86 | 0.01523 | Surface smooth ..... | ..... | x | ..... | ..... | ..... | ..... | ..... | x | x | ..... |
| Aver. | 1.99 | | | | | | | | | | | | | |
| *Commercial Iron and Steels.* | | | | | | | | | | | | | | |
| Commercially pure iron. | | | 0.04290 | Coarse deep pits..... | ..... | ..... | ..... | ..... | x | x | ..... | ..... | ..... | x |
| Open hearth steel..... | | | 0.07320 | Coarse deep pits..... | ..... | ..... | ..... | ..... | x | x | ..... | ..... | ..... | x |
| Copper bearing iron..... | | | 0.03490 | Medium deep pits... | ..... | ..... | x | x | ..... | ..... | x | ..... | x | x |
| Copper bearing steel.... | | | 0.03050 | Shallow pits ..... | ..... | ..... | x | ..... | ..... | ..... | x | x | x | ..... |

A copper-bearing steel, purchased on the open market, was also tested. It contained 0.40 percent manganese and 0.25 percent copper. The loss by corrosion of this steel fits in very nicely with our alloy containing 0.33 percent copper, which it should closely represent.

A copper-bearing iron containing 0.20 percent copper also comes in its proper place on the diagram.

Above 2 percent of manganese the alloys are hard and brittle, so that, because of physical properties, alloys containing more



*A.* *B.*

Fig. 5. Showing Difference in Grain Size.
*A.* 0.48 percent Cu, 0.32 percent Mn × 60.
*B.* 0.58 percent Cu, 1.76 percent Cr × 60.

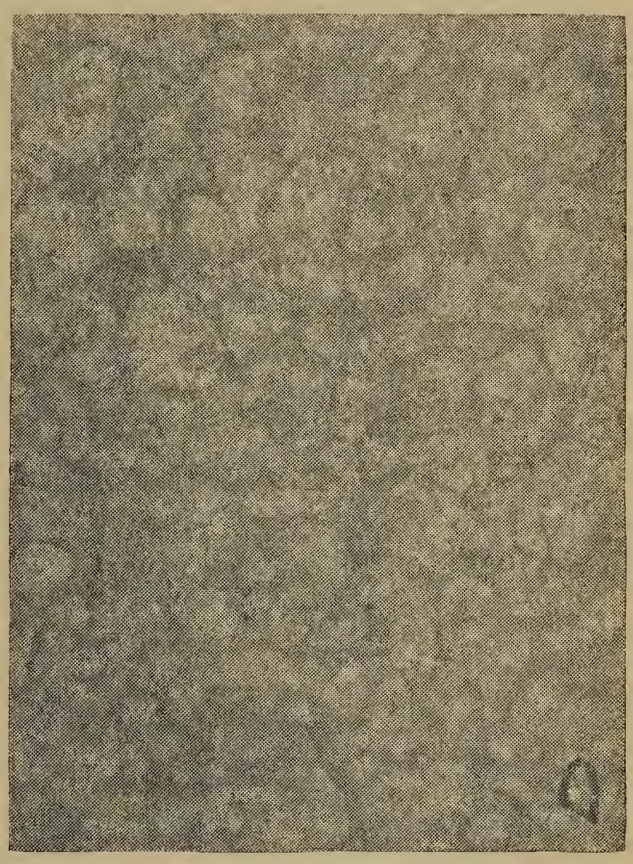
than 2 percent of this element would not find as wide an application in practical work as would alloys containing less than this amount.

*Copper Chromium Alloys.*—The data are given in Table II and in curve form in Figure 4. The chromium copper alloys follow, in general, the behavior of the manganese copper alloys, the chromium having an action similar to that of manganese. Additions of chromium to iron up to about 0.20 percent appear to

increase the amount of corrosion. Above 0.20 percent, additions of chromium increase the resistance to atmospheric corrosion. No explanation for the increase in corrosion when small amounts of chromium are added can be offered. As with manganese copper alloys, the greatest increase in corrosion resistance comes with the first additions of copper, after which further increases in copper are not so effective.

The cause of the influence of copper on the corrosion of iron has never been satisfactorily explained. Mr. D. M. Buck[4] thinks

*C.*



*D.*

FIG. 6. Showing Intergrain Films.
*C.* 3.54 percent Cu, 2.51 percent Mn × 60.
*D.* 3.03 percent Cu, 2.78 percent Mn × 350.

that it acts upon the sulphur, in some way eliminating the bad effects of that element. Dr. W. H. Walker[4] has suggested that copper causes the oxide of manganese and iron to remain in solid solution when they otherwise would be deposited in the inter-grain structure. Burgess and Aston[6] suggest that the iron goes into solution and leaves a coating of copper behind, thus protecting the underlying metal from further corrosion.

The theory here proposed may be called the film or inter-grain

theory of corrosion resistance. It has been known for some years that commercially pure iron is red-short. When a piece of this iron is broken within the temperature range of red-shortness, the fracture is clearly seen to be an inter-grain one. Miller[11] has shown this to be inter-grain, and has ascribed it to an oxide or some other compound of iron. The writers have shown that this red-shortness is due to copper, and that the more copper, the more red-short the iron. This range covers a temperature interval of from about 800° to about 1000° C. during which the inter-grain material is weak or plastic. At a lower temperature it becomes rigid and the iron is no longer red-short. The presence of this inter-grain film, we believe, also gives to commercially pure iron any rust-resisting properties that it may have. Burgess and Aston have shown that electrolytic iron is not rust-resisting, and the remarkable deduction seems almost inevitable that iron only becomes rust-resistant when certain impurities are present in certain amounts. Granting the presence of this inter-grain rust-resisting film, then any element that decreases the grain size or any heat treatment that decreases the grain size will give a further increase in corrosion resistance. This is probably the effect of manganese or chromium (Fig. 5). The presence of manganese or chromium removes the red-short range, but it does not follow that the inter-grain cement is destroyed (Fig. 6). We have obtained some evidence, as yet rather fragmentary, that manganese or chromium does this by lowering the red-short range, so that it exists at a lower temperature, until finally, if enough manganese or chromium be added, the red-short range occurs at a temperature below that at which the inter-grain film becomes weak or plastic. Thus it is possible that with high copper contents the iron might become cold-short.

## SUMMARY.

1. It has been shown that there is a mutual action between manganese and copper in their effect upon the atmospheric corrosion of iron. Copper alone reduces the corrosion of pure iron and, to a still greater extent, the corrosion of steel. This is due to the effect of manganese, which enhances the effect of copper.

2. If manganese is replaced by chromium, the effect is still more pronounced.

3. It is shown that the red-short range in iron, due to the presence of copper, is removed by either manganese or chromium.

4. It is believed that there is some relation between this red-short range and resistance to atmospheric corrosion. Based upon this, the film or inter-grain hypothesis is suggested to explain the corrosion resistance of these alloys.

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## DISCUSSION.

J. A. Aupperle[1] *(Communicated)*: The authors have referred to Dr. A. S. Cushman's[2] discussion of an earlier paper of theirs, where he states that: "copper in either iron or steel is not a prime factor in corrosion resistance under the varied conditions of service and exposure." The authors of this paper seem to have in mind atmospheric tests, while Dr. Cushman refers to tests under varied conditions of service.

The authors have also referred to Kalmus and Blake[3] as finding that copper is beneficial to pure iron, whereas Kalmus and

[1] Chief Chemist, The American Rolling Mill, Middletown, Ohio.

[2] Trans. Am. Electrochem. Society (1916), 30, 390.

[3] Proceedings Am. Inst. Chem. Eng., 1917.

Blake did not have pure iron with copper alone under observation. The authors made tests on iron sheets containing both nickel and copper, but no tests were made on sheets containing iron and copper alone, therefore, no conclusions can be drawn from the benefit of copper when such sheets also contained 0.75 percent nickel.

The conclusions of Kalmus and Blake were based very largely on results of corrosion tests made on small sections which were sawed from castings; these castings having been made in graphite crucibles. The fact that they were made in graphite crucibles changed the analysis so materially that copper-steel alloys instead of copper-iron alloys were produced, on account of the presence of 0.16 percent carbon which the alloys had absorbed from the crucibles.

Reference is also made to work done by O. W. Storey[4], in which the authors say that Mr. Storey found that certain specimens of fence wire that had shown great resistance to atmospheric corrosion contained copper. The authors should also have quoted that Mr. Storey had found some iron wires, Nos. 5a and 18a, which contained only a trace of copper and which were in excellent condition after 25 years' service.

The authors have made the broad statement that the presence of 0.04 percent copper in pure iron causes it to be red-short. This is contrary to our experience, as we have done a large amount of research work on the effect of copper on red-shortness, and have found that if sulphur is low enough, even 0.5 percent copper will not cause the metal to be red-short if the sulphur is below 0.01 percent, even when the manganese is below 0.02 percent.

They claim that the addition of manganese would remove the tendency of the metal to be red-short, which is correct, but it is unfortunate that the authors infer that the manganese combines with the copper, instead of, as is well-known, with the sulphur to form manganese sulphide. They were also the first to make the peculiar claim that in an iron with a high copper content the iron might become cold-short. Statements such as these, that an element can make a metal both hot-short and cold-short, throw

[4] Trans. Am. Electrochem. Soc. (1917), 32, 285.

considerable doubt upon any conclusions which are formed from work containing so much contradictory matter.

We have done considerable work on the effect of chromium, and our atmospheric tests show that up to 0.5 percent chromium, this element increases the rate of atmospheric corrosion; while with very large percentages of chromium, up to 35 percent, the conditions are reversed and practically no corrosion occurs on such alloys. The same remarks apply to the effect of silicon on steel. Small percentages up to 0.5 percent increase atmospheric corrosion, whereas when the metal contains up to 15 percent silicon it is practically rust-proof.

It seems that such sweeping conclusions should not be drawn from atmospheric tests without taking into consideration the many tests which have been made under other conditions.

Very little iron or steel, especially sheet metal, is used without giving it some protective coating, and we have shown that where a galvanized coating on pure iron would last for many years in the Pittsburgh district, all bare grades of iron and steel sheets with or without copper have failed in the Pittsburgh district in three years.

The difference in the behavior of metal when exposed under conditions where water instead of air predominates was referred to by F. N. Speller, Metallurgist of the National Tube Co., in his discussion of Mr. Storey's paper[4]. Mr. Speller said: "In the case of pipe carrying water, for example, we have tried out copper-steel and can confirm everything the author says in regard to that material so far as the outside of the pipe is concerned when exposed to atmospheric conditions, but on the inside the water seems to attack copper-steel as rapidly as ordinary steel without copper. Many tests have been made in service in the same lines carrying ordinary steel pipe, copper-steel and wrought iron, all of which show practically the same results after two or three years' service with respect to the depth of the pitting. While the conditions are nominally the same, that is the presence of water and air, in one case, on the inside of pipe, we have a relatively large proportion of water. On the outside, on the other hand, we have the reverse, viz., a large proportion of oxygen and a very small amount of water. The oxygen in each case is the depolarizing

agent, and the proportion present seems to govern the relative rates of corrosion of these materials."

Mr. Speller has done a large amount of research work on this problem, and I doubt whether copper is purposely added to steel for pipe carrying water. I also know of one company manufacturing wire containing copper, and it is a well-known fact that wire is used to a large extent where air instead of water predominates.

We have done a large amount of research work on corrosion and can confirm what Mr. Speller says about the effect of copper not being beneficial where water predominates. We can even go further than this and say that copper is absolutely harmful where water predominates, especially if the water contains either alkalies or sulphates.

I believe that investigation of corrosion problems made under conditions where water predominates will furnish astonishing data and will emphasize the danger of drawing general conclusions as to the corrosion resistance of a metal subjected to atmospheric conditions only.

E. A. Richardson: In electrolytic iron containing 0.006 per-cent sulphur, the presence of small traces of copper makes the iron short. In regard to these instances that have been cited about the rust-resisting properties of iron, I would like to ask the previous speaker what his opinion is, how he reconciles this with the results of the tests of the American Society for Testing Materials?

J. A. Aupperle: I should like to ask Mr. Richardson how much hydrogen and silicon the electrolytic iron contained which he found to be red-short.

E. A. Richardson: We have really no analysis on the amount of hydrogen contained, but this material was melted at perhaps 1,-700°C.

J. A. Aupperle: I believe it is extremely dangerous to draw conclusions from insufficient data, and I think that the evidence should be carefully considered before any claims are made that copper renders the iron red-short.

The electrolytic iron referred to by Mr. Richardson may have

taken up a small percentage of some other element such as silicon which has an important bearing upon the subject, as it is a well-known fact that the addition of a small amount of silicon to pure iron made in open-hearth furnaces makes the metal red-short.

In reference to reconciling the facts of the Pittsburgh tests with statements that I made that copper is of no benefit to steel, I distinctly stated that I referred to tests in general and not to atmospheric tests alone. For instance, it is a recognized fact that copper confers no benefit whatever where water instead of air predominates. My conclusions therefore were from tests in general and not one specific atmospheric test.

J. W. RICHARDS[5]: In view of the remarks of Mr. Aupperle I think it would be advisable if the author would correct the title to read, the effect of these elements on the atmospheric corrosion of iron and steel, since that was the subject treated in the paper. There is a statement also at the top of page 232, "It has been known for some years that commercially pure iron is red-short." Is that correct? Commercially pure iron is not red-short. And a little further down: "The writers have shown that this red-shortness is due to copper." But the iron does not always contain copper. I think the above statement means: "It has been known for some years that commercially pure iron which contains copper is red-short."

L. T. RICHARDSON: As far as I know, commercially pure iron has always contained copper in small amounts, and I think the authorities have always stated that it was red-short and should not be worked within this range.

J. W. RICHARDS: Would you say that commercially pure iron not containing copper is red-short?

L. T. RICHARDSON: I do not think so, but I think commercially pure iron is never free from copper.

J. W. RICHARDS: I do not think it always contains copper.

COLIN G. FINK[6]: I have a remark to make in regard to the method of expressing corrosion loss. In reviewing papers on corrosion it is very unfortunate that each author selects his own

[5] Prof. of Metallurgy, Lehigh University, Bethlehem, Pa.
[6] Head of Laboratories, Chile Exploration Co., New York City.

unit, and I think it is up to the Society to standardize on one particular unit; the unit of loss in grams per square centimeter per year is a very convenient one. If the Society would standardize on that or a similar unit, it would assist readers a good deal. The curves in the paper before us are expressed in three or four different units. In regard to Mr. Aupperle's statement as to the effect of copper on red-shortness, I think we can reconcile Dr. Richards' remarks and Mr. Aupperle's. We know that the solubility of copper in iron varies considerably, depending upon the impurities such as carbon or sulphur, present in the iron. It is conceivable that we may have copper present and yet the metal not be red-short; two samples may contain the same percentage of copper, but in one case the copper is in solution and in the other in the interfaces.

O. W. Storey[7] *(Communicated)*: The data given in this report are very interesting, and a distinct contribution to the literature on the corrosion of iron. It will be very interesting to know what the results are, after several more years of corrosion of the specimens listed in this paper. Since the quantitative data cover only the corrosion during a period of 8 months, it may be possible that the results will have materially changed during a much longer period. Mr. D. M. Buck has shown that the rate of corrosion of the alloy steels changes materially after a first preliminary corrosion. It would seem from this that the quantitative data of this paper show the rate of corrosion of these alloys covering the first losses. I do not believe that any material change will come in the relative rates of the low and high copper alloys, but there will be some difference among the high copper alloys themselves, that is, it may be possible that the high copper alloys will not show as much difference among themselves for a longer period of time, or on the other hand, they may show a much greater difference.

The figures show that when copper is added to pure iron, there is a decrease in the amount of corrosion until about 1 percent copper is reached, when the additional increase of copper does not seem to show any additional benefit. The results of the tests by the American Society for Testing Materials would seem to

[7] Metallurgical Engineer, C. F. Burgess Lab., Madison, Wis.

indicate that the data given in this paper cannot be applied to ordinary steels. A study of the figures given in the report of Committee A-5 during the 23rd Meeting of the American Society for Testing Materials, June 22-25, 1920, seems to show that there is another element influencing the rate of corrosion of steel besides copper or manganese. In groups *B, H, I, K, M, T,* and *Z* the amount of manganese is practically the same. The copper content does not vary much, yet in some of these cases the sheets have gone completely, while in other cases they seem to be in excellent condition. The only element which seems to have an influence is the phosphorus. The low phosphorus sheets among the open-hearth steels have failed, while the high phosphorus sheets apparently are in good condition. The only exception is group *H,* and this group shows a higher phosphorus content than any of those which have failed, and the report of the Committee also shows it to be corroding faster than those higher in phosphorus. All of the pure irons are low in phosphorus, and all of these pure iron sheets are failing. Those higher in copper, however, fail at a slower rate than those low in copper. It is apparent, therefore, that while copper is the main influence, some other element or condition has a secondary influence. From the data given in the Committee Report, phosphorus would seem to be the secondary element, rather than manganese. More data are required, however, to determine exactly whether the influence of the phosphorus is of benefit. Buck has shown that steel containing high phosphorus is more resistant to corrosion than one containing low phosphorus, although he believes that sulphur is the cause of the rapid corrosion of steel sheets low in copper.

While the data of this paper leave no doubt as to the beneficial influence of either manganese or chromium when added to copper-irons, it does not necessarily follow that high manganese or chromium will have the same beneficial influence on all of the steels high in phosphorus and other elements.

It has been suggested to the writer that the high phosphorus forms a copper phosphide, which forms an inter-grain cement, which is especially resistant to corrosion. This idea was taken from the use of phosphorus in phosphor bronzes.

J. A. Aupperle *(Communicated Jan. 25, 1921)*: I have since

had the opportunity to study the effect of copper when added to electrolytic iron; the alloys of iron and copper being made in an Arsem furnace. These alloys analyzed as follows:

| | Ingot No. 31 Percent | Ingot No. 32 Percent | Ingot No. 33 Percent | Ingot No. 34 Percent |
|---|---|---|---|---|
| Silicon | 0.031 | 0.060 | 0.040 | 0.070 |
| Sulphur | 0.015 | 0.014 | 0.018 | 0.017 |
| Phosphorus | 0.004 | 0.004 | 0.004 | 0.007 |
| Carbon | 0.015 | 0.015 | 0.010 | 0.018 |
| Manganese | 0.006 | 0.015 | 0.024 | 0.024 |
| Copper | 0.050 | 0.090 | 0.336 | 0.504 |
| Oxygen | 0.040 | 0.020 | 0.023 | 0.021 |
| Carbon monoxide | 0.018 | 0.011 | 0.013 | 0.012 |

In my discussion I assumed that the presence of silicon in the vacuum furnace melted iron may have been responsible for the red-shortness noticed by the Messrs. Richardson. These alloys, as seen from the above analyses, had absorbed silicon from the lining.

A careful forging test was made on each of these alloys by working them back and forth over an anvil at temperatures ranging from 1100° to 500°C. This is a very satisfactory test for determining whether the metal is red-short. The alloys experimented with ranged from 0.05 to 0.5 percent copper, and it was found that there was absolutely no red-shortness in the higher copper alloys, and only a slight red-short tendency was observed on the ingot containing 0.05 percent copper. This alloy incidentally contained only a trace of manganese, which undoubtedly caused the metal to be slightly red-short. The important factor, however, is that we could not substantiate the Messrs. Richardson's claim that the higher the copper the more red-short the metal up to a half percent copper.

These tests also have shown that the silicon in these alloys made in an Arsem furnace does not have the same influence as silicon added to pure iron made in open-hearth furnaces.

These tests confirm what I claimed in my discussion that copper does not cause red-shortness; this phenomenon is largely dependent upon the sulphur content.

L. T. RICHARDSON (*Communicated*): It is rather difficult to answer Mr. Aupperle's discussion since there is so much destructive criticism and so little constructive criticism in it. Most of his remarks are not germane to the paper in question.

He objects to our quoting Dr. A. S. Cushman as follows: "Copper in either iron or steel is not a prime factor in corrosion resistance under the varied conditions of service," saying that Dr. Cushman referred to "varied conditions of service." Is not atmospheric corrosion one of the varied conditions of service? Is it not the most important condition of service?

Mr. Aupperle also objects to our referring to the work of Kalmus and Blake because the material tested by them contained about 0.15 percent carbon which was obtained from the "graphite crucible used" according to Mr. Aupperle. Kalmus and Blake made their alloys by melting A.R.M.Co. iron and the alloying elements in a clay crucible in a gas-fired furnace. According to the authors, the small amount of carbon present in the alloys was absorbed from the gas. Apparently Mr. Aupperle is mistaken in his reading of this paper by Kalmus and Blake. Kalmus and Blake's alloys were substantially pure iron with copper added and they came to the conclusion that copper added to pure iron increased the resistance to corrosion.

The same gentleman also objects to our quoting a paper by O. W. Storey. Storey found from a number of specimens of fence wire that all specimens of steel wire that were rust-resisting contained copper. Two pieces of wrought iron were also found that appeared to be rust resisting. These two wires were wrought iron and not pure iron as might be surmised from Mr. Aupperle's discussion. Moreover, Mr. Aupperle forgets to state that one of these wires (5a) contained large pits and that the other (18a) was originally painted. Furthermore, our paper does not deal with the corrosion of wrought iron so that there is no reason why we should quote the findings of Mr. Storey on a material foreign to the subject of this paper.

Mr. Aupperle's discussion of Mr. Speller's work on the corrosion of iron pipe has nothing to do with atmospheric corrosion and does not belong in a discussion of this paper.

The argument that sheet iron is usually given some protective

coating such as paint or galvanizing is true. At the same time protective coatings do not last forever and are often broken or removed in spots, so that the more rust resisting the base metal the better.

Mr. Aupperle's statement that all grades of uncoated iron and steel, with or without copper, have failed in three years in the Pittsburgh district, is too indefinite to mean anything. What was the gauge of the sheets? If the sheets were thin enough all might fail in one year or if thick enough all might last 50 years. The official test of the American Society for Testing Materials on 22-gauge uncoated sheets at Pittsburgh shows all of the copper-bearing steels still intact at the end of three years and all of the copper-free steels and commercially pure irons (A.R.M.Co. Iron included) to have failed. It is rather surprising that Mr. Aupperle, as a member of the Corrosion Committee of the American Society for Testing Materials should make such a misstatement as the above.

Much of Mr. Aupperle's discussion is taken up with his objections to the statement that copper causes iron to be red-short, with which he, as usual, disagrees. He begins by saying that copper does not make commercially pure iron red-short provided that the sulphur is below 0.01 percent. We would infer from this that if sulphur is above 0.01 percent that copper does cause red-shortness. Mr. Aupperle thinks that the addition of silicon to electrolytic iron may cause it to be red-short, yet he does not explain why the same iron free from copper is not red-short. Later on, in some of his own work, he finds that silicon does not cause red-shortness in electrolytic iron. His conclusion that small amounts of silicon added to commercially pure iron causes it to be red-short is not warranted because such iron is never free from copper, and is still red-short when free from silicon. He reports the results of forging tests on four alloys, numbers 31-34, one of which was red-short. These alloys were tested over an anvil at temperatures ranging from 1,100° C. to 500° C. It is unfortunate that Mr. Aupperle did not state the results of his tests above 1,100° C. and also tests of electrolytic iron free from copper. Alloy 31 containing 0.015 percent sulphur and 0.05 percent copper is red-short. If sulphur is the cause of this red-shortness, then elec-

trolytic iron containing 0.015 percent sulphur and free from copper should be red-short. Of all the published data on forging tests of electrolytic iron, we know of no case where this has been found.

Our paper deals primarily with ordinary commercial irons and not with iron of extreme purity such as electrolytic iron. In the present state of our knowledge, we do not believe that with such irons red-shortness can be attributed to sulphur. In ordinary iron where sulphur is relatively high and manganese low the iron is red-short due to an intergrain film of iron sulphide. When manganese is added, insoluble MnS is formed. It is known that MnS is produced because it can be detected and identified under the microscope.

When it comes to traces such as are present in Mr. Aupperle's electrolytic iron, phase relations and mass action may change conditions entirely and other conditions may result. With commercial irons we are still convinced that copper causes red-shortness and that this may be eliminated by the addition of manganese. When one speaks of iron or steel one means iron with the usual impurities, such as P, S, C, etc. If now it is found that copper added to such an iron produces red-shortness, we believe that we are perfectly accurate in saying that copper is the cause. This follows from the definition of iron. It matters not which impurity combines with copper, assuming that it does, to cause red-shortness. If these impurities are usually present in the iron, then it may be said that copper is the primary cause. The Navy Department finds[8] that when copper is present in steel a higher manganese content is needed to prevent red-shortness.

Prof. Richards questions our statement that commercially pure iron is red-short and that it always contains copper. We have never seen an analysis of commercially pure iron that does not show 0.02 to 0.05 percent of copper. This fact is so readily admitted even by the manufacturers themselves, that there is no question as to its truth. The same is true as to the red-shortness of commercially pure iron. The trade catalogues of the manufacturers admit that their product is red-short and some of the claims of the Carnahan patents on commercially pure iron are based upon this property.[9]

[8] Chemical and Metallurgical Engineering, Feb. 11, 1920.

[9] French patent 467,332; Norwegian patent 27,684; U. S. patent 1,090,115; British patent 28,571.

Dr. Fink's criticism is constructive and confirms our contention that copper need not be present as metallic copper but perhaps as some compound of copper.

It is very probable that other elements such as phosphorus and sulphur affect the corrosion of copper-bearing iron and steels and it is not surprising that Mr. Storey should find examples of this in the data of the corrosion test of the American Society for Testing Materials. In fact, it would be strange if such differences did not occur. It is too early yet to draw detailed conclusions from this test and only general ones are warranted. The *Z* test seems to indicate that copper-bearing Bessemer steels (high phos.) are no better than copper-bearing open-hearth steels (low phos.). Other work by Buck also indicates this. We still believe that a high manganese, high copper steel would be decidedly more rust-resisting than any material in the official test as reported by Mr. Storey.

*A paper presented at the Thirty-eighth General Meeting of the American Electrochemical Society, in Cleveland, October 2, 1920, President Landis in the chair.*

# THE SOLUTION OF METALS IN ACIDS AS RELATED TO CORROSION.[1]

By William D. Richardson.[2]

## INCONSISTENCIES OF THE ELECTROCHEMICAL SERIES.

The inconsistencies shown by the electrochemical series of the metals as regards solution in acids and corrosion, which may be defined as the solution of metals in water or electrolytes, in the presence of air, are numerous. For one cause or another, the tendency of many metals to go into solution in acids is quickly slowed down, and thereafter solution stops altogether or the rate becomes so low as to be negligible. To give a concrete example, aluminium, from its position in the electrochemical series, ought to dissolve readily and rapidly in acids, and corrode rapidly in water, as compared to copper. Nevertheless, under many conditions the rates for the two metals are close together, and under some conditions the rate is higher for copper than for aluminium, whereas iron, which occupies a position about midway between the two, as a rule dissolves more rapidly in both acid and neutral media than either.

Different investigators give the different metals somewhat different positions in the electrochemical series, due perhaps primarily to differences in the experimental conditions maintained, but the following table represents, generally speaking, the consensus at present on the subject. It should be noted, in passing, that the differences in experimental conditions referred to are of the same kind and of the same order which also affect the various metals under corrosion conditions and when dissolving in acids.

[1] Manuscript received August 9, 1920.
[2] Chief Chemist, Swift & Co., Chicago, Ill.

*Electrochemical Series of the Elements.*

| Non-metals | Metals | | |
|---|---|---|---|
| Fluorine | Osmium | Hydrogen | Magnesium |
| Oxygen | Rhodium | Lead | Calcium |
| Chlorine | Iridium | Tin | Strontium |
| Bromine | Gold | Nickel | Barium |
| Iodine | Platinum | Cobalt | Lithium |
| Sulphur | Palladium | Iron | Sodium |
| Phosphorus | Silver | Cadmium | Potassium |
| Selenium | Mercury | Zinc | Rubidium |
| Nitrogen | Copper | Manganese | Cæsium |
| Boron | Arsenic | Chromium | |
| Carbon | Bismuth | Aluminium | |
| Silicon | Antimony | | |

## RELATION OF SOLUTION IN ACIDS TO CORROSION.

It has been realized, ever since the electrolytic theory of corrosion was put forward, that there must be a rather intimate relation between the phenomena of solution of metals in acids and corrosion itself, and numerous attempts have been made to develop a so-called accelerated corrosion test in acids, particularly for various kinds of iron and steel, which would run parallel to actual corrosion tests under service conditions, for the respective metals. All such attempts have proven failures, and the course of corrosion has been found to be different from that of solution in acids, so that the latter could not be made to serve as an index of the former. Thus, not only do the metals dissolve in acids in a different order from that which might be expected from the electrochemical series, but they follow a still different order of corrosion, or, rather, of corrodibility.

Some of the anomalies of the electrochemical series were discussed in a previous paper.[3] In that paper certain experiments were reported which showed a comparison between the corrodibility of aluminium and copper under various conditions for 950 days. Under most of the conditions the two metals corroded at approximately equal rates, but where there was a difference, the copper corroded faster than the aluminium. Neither corroded at a rate comparable to that of iron under the same circumstances.

[3] Experiments on the Corrosion of Iron and Steel. Proc. Am. Inst. Chem. Eng. (1920).

## PRIMARY ESSENTIALS FOR CORROSION OR SOLUTION IN ACIDS.

These essentials have been stated many times, in different forms, by various writers, but it seems advisable to re-state them here, since it appears that they have not become altogether matters of common chemical knowledge. They are:

1. The dissolving metal, usually of relatively high solution tension.

2. A voltaic couple consisting of a metal anode (the dissolving or corroding metal) and a cathode, which may be metallic or consist of impurities found in or on the metal, graphite, metallic oxides, carbonates or other compounds.

3. An electrolyte containing hydrogen or metal ions.

4. For continued solution or corrosion the cathode must be of such a character that hydrogen is readily released therefrom so that polarization is avoided, or the hydrogen must be removed in the form of water by oxidation. In this reaction the cathode material may assist as a catalyst.

5. Corrosion is assisted by precipitation of the first products formed, for example, as oxides, hydroxides or carbonates, and suitable reactions are brought about by the presence of oxygen, carbon dioxide, etc. In this manner the osmotic pressure of the dissolving metal is kept low.

These primary essentials and various retarding factors in corrosion are discussed below.

## THEORETICAL RATE AND ACTUAL RATE.

On theoretical grounds it may be assumed that corrosion of metals in water and solution of metals in acids tend to go forward at rates proportional to the solution tension of the metal and the hydrogen ion concentration of the electrolyte, in the absence of disturbing influences, but, as a matter of fact, solution and corrosion rarely proceed at this theoretical rate. Under nearly all conditions, one or another influence makes itself felt in such a way as to promote or retard the action, and it is necessary in each individual case, if the rate of solution or corrosion is to be determined or explained, to ascertain the positive and negative accelerating effects of a number of different factors. Neglecting

temperature and pressure, some of the principal influences which affect the rate of solution and corrosion are discussed below in detail. Knowing the effect of each of these influences, there can be no doubt that solution in acids and ordinary corrosion could be completely correlated.

## INFLUENCES AFFECTING THE RATE OF SOLUTION.

Of all the influences which act to retard or prevent the natural tendency of metals to go into solution in acids or water (corrosion) from being realized in proportion to the tendency (solution pressure), the most important are two in number, and in this paper I call them, considering both in the broadest sense, the "hydrogen influence" and the "oxygen influence," respectively. Each of these is greatly modified in its action by the other influences enumerated, such as the condition of the surface, the nature of the products of solution or corrosion, the presence of catalysts, oxygen or oxidizing agents, chlorine or other halogens, and the composition and impurities in the metals.

### *Solution Tension.*

The solution tension of a metal which determines its position in the electrochemical series, one might suppose would be a dominant factor in determining the rate of solution in an acid, as well as the rate of corrosion. Abundant evidence shows that it does neither of these things. It represents only a tendency to go into solution, a tendency which under the ordinary conditions of experiment is so quickly brought to a halt by various influences, on the other hand so readily accelerated by catalysts, and so seldom realized, that the actual order of corrodibility and solution of metals is quite at variance with the order in the electrochemical series. It is so easily over-balanced by polarization with hydrogen, by the influence of oxygen with or without the resultant formation of a passive layer, by the effects of impurities in the metal and by the influence of the products of corrosion, that it is rarely the determining factor.

### *The Hydrogen Influence.*

By this term are designated all those effects which result from the action of hydrogen released against a metal surface by electro-

lytic action in the presence of an acid or water (corrosion). When a piece of relatively pure iron such as ingot iron is placed in dilute sulphuric or hydrochloric acid, very little visible action ensues. There is a tendency for the iron to go into solution in the acid, thereby releasing the hydrogen. This hydrogen is adsorbed or occluded by the iron and the action soon comes to a halt or proceeds at an extremely low rate. If in place of a non-oxidizing acid, however, an oxidizing acid such as nitric is used so that any hydrogen released is quickly oxidized to water and polarization thus prevented, the action goes forward at an enormously accelerated rate. The hydrogen occluded by the iron when non-oxidizing acid is used can be released by suitable physical means. Such a piece of iron can be shown to be electro-positive to a piece of untreated bright iron of the same sort, carefully cleaned by means of emery cloth, when the two are placed in a simple cell containing neutral salt and the circuit completed through a milli-voltmeter. When a catalyst such as a small amount of platinum salt is added to the non-oxidizing acid and a piece of pure iron immersed therein, solution goes on rapidly with evolution of hydrogen. Apparently no considerable polarization results. Cast iron too dissolves readily in non-oxidizing acids, thus suggesting the presence of catalysts among the impurities. Graphitic carbon might act as the catalyst in this case by serving as a cathode and releasing the hydrogen ions migrating to it.

Yet it cannot be doubted that such closely related phenomena as the solution of metals in acids and the corrosion of the same metals in water in the presence of air should show a certain degree of similarity and that the one type of reaction should be comparable with the other either directly or after taking into consideration the differences in the two cases. Both should be susceptible of explanation on the basis of the electrolytic theory and the electrochemical series.

In a previous paper an attempt has been made to work out a tentative correlation between the corrosion of iron and steel in the presence of abundant oxygen and a limited supply of oxygen and the solution of the same metals in oxidizing acids and non-oxidizing acids.[4] In that paper the results show that pure iron

[4] Rates of Solution of Iron and Steel in non-oxidizing and oxidizing acids. Proc. Am. Inst. Chem. Eng., Montreal Meeting (1920).

dissolves relatively less in non-oxidizing acids and relatively more in oxidizing acids than cast metals, such as grey cast iron and semi-steel, and that a similar relationship holds for the corrosion of these metals in the presence and absence of oxygen. A suggested basis for an index of corrosion based on these observations is made the subject of a succeeding paper. Many more correlations of this sort remain to be worked out for various other metals besides iron and steel.

Thus it is seen that when a metal dissolves in non-oxidizing acids the resultant hydrogen may be adsorbed or occluded by the metal surface resulting in polarization and a slowing down of the rate of action; or the hydrogen may be released not on surfaces of the metal going into solution, but on cathodes formed by impurities in the metal, such as graphitic carbon in cast iron or catalytic metals. In this way polarization does not so readily supervene and solution goes on at a fairly rapid rate; or in the presence of oxygen, an oxidizing agent, or an acid, the hydrogen may be oxidized to water at the cathodes as rapidly as it is produced and in this way removed from the sphere of action altogether. Metallic catalyzers probably act in both these ways, in the first instance reducing the over-voltage and promoting the release of hydrogen and in the second catalyzing the hydrogen-oxygen reaction.

### *The Oxygen Influence.*

Oxygen affects the solution of metals in acids and in water in a variety of ways, generally in such a manner as to accelerate the action, but under other circumstances in such a way as to prevent action altogether. Its accelerating action is due usually to its oxidizing effect on hydrogen as indicated above or to its ability to produce cathodic materials by acting on the first products of corrosion, for example, by the production of rust in the corrosion or iron. The passive layer on a metal surface may also be classified as an oxygen effect, and when such a layer is formed, solution is prevented altogether. The influence of oxygen is evidenced in a number of different ways:

1. By condensation on the metal surface with or without the production of a film of oxide so as to produce cathodic areas;

2. In bringing about the hydrogen-oxygen reaction so as to remove hydrogen as water;

3. By oxidizing the first product of corrosion, for example, ferrous compounds to ferric;

4. By producing passivity.

The third action may be termed in general a precipitating action as distinguished from a mere oxidizing effect. Ordinary oxidizing agents and oxidizing acids influence the solution of metals usually by oxidizing the hydrogen produced and by preventing polarization. Generally speaking, the oxygen influence is one of the most important if not the most important influence of all in determining the course of corrosion and the solution of metals in acids.

*The Carbon Dioxide Influence.*

It has been held by several investigators that an acid is an essential prerequisite to corrosion. This is true in the sense that hydrogen ions or the equivalent are necessary. Advocates of the acid theory of corrosion have claimed that under ordinary circumstances the carbon dioxide of the air, through its ability to produce carbonic acid, furnished the necessary hydrogen ions for corrosion. This point has been the subject of much controversy and some of the experiments brought forward to prove the necessity for the presence of carbon dioxide in atmospheric corrosion have been criticized and the results questioned. The literature on the subject is voluminous.[5] So far carbon dioxide or carbonic acid acts as an acid, but it may also act as a precipitating agent, removing metallic ions from solution and keeping the osmotic pressure of the dissolving metal at a low point. To this extent and in this way it acts in a manner similar to oxygen when the latter assumes a precipitating role. Thus in the corrosion of lead for the manufacture of white lead, carbon dioxide acts primarily as a precipitating agent and the basic lead carbonate precipitated undoubtedly forms cathodes, thus accelerating the corrosion just as rust accelerates the corrosion of iron. For, as might be expected, the accelerating influence of rust on the corrosion of iron is not an unusual or isolated phenomenon. The products of corrosion of all the

[5] G. T. Moody, Proc. Chem. Soc. (1903), **19**, 157, 239; (1907), **23**, 84; Trans. Chem. Soc. (1906), **89**, 720; W. R. Whitney, J. A. C. S. (1903), **25**, 394; H. A. D. Jowett and E. Goulding, Trans. Chem. Soc. (1905), **87**, 1548; W. H. Walker, J. A. C. S. (1907), **29**, 1251; J. A. N. Friend, J. Iron Steel Inst. (1908), **1**, 5; W. H. Walker, J. Iron Steel Inst. (1909), **1**, 69; B. Lambert and J. C. Thomson, Trans. Chem. Soc. (1911), **97**, 2426; J. A. N. Friend and J. H. Brown, Trans. Chem. Soc. (1911), **97**, 1302.

commoner metals such as aluminium, zinc, nickel, lead, copper, etc., all act as accelerators of corrosion when discontinuous and as protectors against corrosion when they form continuous coatings adherent to the respective metals. They are all electro-negative in character to their parent metals. In some cases the products of corrosion tend to act in a protective capacity rather than otherwise, and in other cases more in the capacity of accelerators of corrosion. For the most part the latter action appears to predominate in the case of iron and the common varieties of alloys of iron. When the first product of corrosion is capable of oxidation to a higher oxide or compound, the electro-negative character of the product is increased and hence also its ability to act as an accelerator. This is true of iron. The corrosion of manganese has not been thoroughly studied, but such a study should yield important results.

### *The Cathode Influence.*

In the absence of definite cathodes on a metal surface, corrosion and solution in acids proceed slowly. In general polished surfaces and very pure metals do not dissolve in non-oxidizing acids or corrode as rapidly as less pure metals and unpolished surfaces. When suitable cathodes are present they may act in two ways. First as regions and points to which hydrogen ions migrate, on which hydrogen is deposited, and from which it is released more or less readily (usually more readily) than from the metal undergoing solution or corrosion; again cathodes may act in such a way as to promote or accelerate the hydrogen-oxygen reaction by which hydrogen is oxidized to water and so removed from the sphere of action. The last effect naturally can occur only in the presence of oxygen or an oxidizing agent. Cathodes sometimes consist of oxides of the metal undergoing solution as, for example, ferric oxide formed when iron rusts, or an insoluble metallic salt as carbonate or basic sulphate.

### *The Action of Catalytic Materials.*

All of the substances (or influences as I have termed them in this discussion) which affect the theoretical rate of solution of corrosion in a positive or negative manner so as to accelerate or retard that rate may be considered as catalyzers; but in what fol-

lows the results of experiments with metallic catalyzers are particularly discussed.

A reasonable explanation of the action of catalyzers such as platinum and the more noble metals generally in promoting solution in acids and corrosion, is that they are deposited at close intervals over the surface of the reacting metals (producing what may be visualized as a finely mottled effect) so as to form catalytic cathodes to which hydrogen migrates and from which it is released more readily than from the dissolving metal itself. They may also act as accelerators of the hydrogen-oxygen reaction when oxygen or an oxidizing agent is present.

In dealing with the effect of metallic catalyzers on solution and corrosion there are several substances present as impurities in the metals or evolved therefrom under the action of acids or during corrosion which may affect the accelerating or retarding influence of the catalyzers themselves. These substances are especially sulphur, arsenic, hydrocarbons and other catalyzer poisons. When comparing the accelerating effect of catalyzers on relatively pure samples of iron and impure samples such as cast iron, naturally very much larger amounts of catalyzer poisons are evolved from the latter than from the former, and in a complete study of the subject it would be necessary to eliminate these influences one by one in order to ascertain the basic facts. In the present studies the metals have been taken as ordinarily produced and experimented with in their original condition, thus unfortunately involving two unknown factors, the catalyzer and the poison, in the same experiment. Nevertheless some conclusions can be drawn from even these experiments.

### *The Halogen Influence.*

Byers and others have shown that passivity phenomena can readily be called into existence by the electric current acting in electrolytes containing oxygen salts and acids, but not in halogen acids or salts or when halogens are present. Passivity phenomena are closely related to the oxygen effect which shows itself in connection with the solution of metals in acids and corrosion. Therefore, the solution of metals in halogen acids, such as hydrochloric, is apt to take a different turn from that in oxygen acids even

when the oxidizing effect of these is rather low. No attempt is made here to offer any hypothesis explaining the action of chlorine at anodes during the solution of metals in acids, but the facts appear to warrant the statement that chlorine at an anode has a deoxidizing effect or prevents the formation of the oxide film associated with any passivity phenomena and the related solution and corrosion phenomena. Aluminium shows a different electrolytic behavior in hydrochloric from that in nitric or even in sulphuric acid (see below Behavior of the Aluminium-Iron Couple). The solution of aluminium in hydrochloric acid is catalyzed by platinum but not in sulphuric acid. Here we see apparently the oxygen and chlorine effects acting in contrary directions.

## BEHAVIOR OF VARIOUS COUPLES.

Considerable information can be obtained concerning the behavior of metals in acids and during corrosion from their behavior as electrodes in a simple cell containing water, acids or salts, the electrodes being separated by a diaphragm. In the experiments described and discussed below a sensitive Weston voltmeter was used as the indicating instrument. In experiments of this kind the hydrogen influence, the oxygen influence, the halogen influence, the effects of the products of corrosion and others can be studied qualitatively. Quantitative experiments are not included in what follows.

## THE HYDROGEN INFLUENCE ON PURE IRONS, CAST IRON, COPPER AND ALUMINIUM.

When a piece of relatively pure iron such as ingot iron is soaked in dilute sulphuric acid for some time or for a shorter time in stronger acid, say 10 percent, as is well-known, hydrogen is absorbed and the metal becomes more or less brittle. This hydrogen can be removed by physical means. When such a piece of iron is tested in a simple cell containing water acidulated with dilute sulphuric acid against a similar piece of iron thoroughly cleaned and preferably dipped into boiling water before the test, the acid-soaked iron shows by a momentary deflection of the galvanometer needle that it is positive to the untreated iron. Soon, however, the needle is strongly deflected in the opposite direction and remains

so deflected. If, however, instead of using water acidulated with sulphuric acid, a neutral salt solution such as NaCl or $Na_2SO_4$ is used, and the iron soaked either in hydrochloric or sulphuric acid, it is positive to untreated iron and remains so for a long time.

Grey cast iron or semi-steel soaked in either sulphuric or hydrochloric acid and tested against similar iron in the simple cell described above, containing either dilute sulphuric or hydrochloric acid or neutral salt, is positive to the untreated cast iron. When such a piece is tested in dilute acids, the cast metal presents the peculiar phenomenon of evolving hydrogen gas at the anode, that is, from the acid-soaked plate, while the galvanometer needle indicates that the current is passing from this plate to the untreated plate. This is similar to the action of zinc in a simple cell containing acid with copper as the cathode.

Copper soaked for a long time in dilute sulphuric acid is always decidedly positive to untreated copper when tested in dilute sulphuric acid.

Aluminium when soaked in dilute sulphuric acid and tested in dilute sulphuric acid is always negative to an untreated aluminium electrode. The same holds true for aluminium soaked in HCl and tested in neutral NaCl solution. Since aluminium is so sensitive to oxygen it might be expected that the acid-soaked metal, if, in fact, it contains adsorbed hydrogen, would be positive to untreated aluminium. This point has not been fully investigated, and it may be that the acid-soaked metal in fact retains basic sulphate or some form of oxide layer on its surface which dominates the action and directs the current to it as a cathode. This behavior should be considered in connection with the action of the aluminium-iron couple and the solution of aluminium in acids with platinum as catalyzer.

Langdon and Grossman, in a paper presented at the last meeting of this Society,[6] found that the pickling of carbon steels in both hydrochloric and sulphuric acids of twice normal strength produced about the same reduction in "stress period," in other words, about the same embrittling effect, but that "with nitric acid practically no reduction in the stress period of rods was produced." Oxidizing agents, such as twice normal nitric acid and molar

[6] The Embrittling Effects of Cleaning and Pickling Upon Carbon Steels. Trans. Am. Electrochem. Soc. (1920), 37, 543.

potassium bichromate, "exerted no measurable effect upon the brittleness." Interpreted in terms of hydrogen occlusion, these results mean that approximately the same amount of hydrogen is occluded from both HCl and $H_2SO_4$, but that no hydrogen is occluded from $HNO_3$. They did not experiment with either pure irons or cast irons.

### OXYGEN INFLUENCE: BEHAVIOR OF THE ALUMINIUM-IRON COUPLE.

Instructive light on the oxygen influence in general and the relationship of oxygen to aluminium in corrosion and solution phenomena in particular is afforded by the behavior of aluminium toward iron in a simple cell containing the dilute acids.

A clean piece of nearly pure iron and a clean piece of nearly pure aluminium dipped into dilute sulphuric acid in a cell furnished with a porous asbestos diaphragm, the terminals being connected through a sensitive voltmeter, present the curious spectacle at first of the iron being positive to the aluminium. If allowed to remain at rest for some time the aluminium may become positive to the iron, but exposure to the air again causes it to become negative to the iron. If, on the other hand, the iron is exposed to the air, the iron may become negative to the aluminium. This experiment again shows the overbalancing effect of the oxygen or oxide electrode in the case not only of iron, but of a metal such as aluminium with a much higher solution tension than iron.

On the other hand, if copper is substituted for the iron the aluminium is invariably positive to it.

In dilute nitric acid the iron is also positive to the aluminium. If the iron is exposed to the air for a short time it becomes electrically neutral or even slightly electro-negative to the aluminium. On the other hand, if the aluminium is exposed to the air it becomes more strongly electro-negative to the iron.

If both pieces are simultaneously exposed to the air and dipped simultaneously into dilute nitric acid, the aluminium is electrically neutral or slightly electro-negative to the iron. After immersion in the dilute nitric acid for a short time the difference in potential increases, the iron being electro-positive to the electro-negative aluminium. If the couple is allowed to stand for some time, gradually the aluminium becomes electro-positive to the iron. When

this condition has been reached the iron pole may be lifted into the air and replaced in the cell without greatly changing the deflection of the voltmeter needle. If now the aluminium pole is removed from the cell and brought into contact with the air for a short time, the aluminium becomes strongly electro-negative to the iron, stronger in fact than during the first few moments of the experiment, nor can the current be again reversed in any reasonably short time by leaving the aluminium pole in the cell and exposing the iron pole to the air. These experiments show of how little moment is solution tension in corrosion phenomena in the presence of air, oxygen, or an oxidizing agent, and how easily overbalanced the relatively high solution tension of aluminium is by oxygen or an oxide. By preference the current flows from iron with low solution tension to aluminium with high solution tension, although iron itself is capable of forming an oxide which is strongly electro-negative to iron itself. Evidently, however, oxygen on an aluminium surface or an oxide of aluminium, if this is formed, is sufficiently electro-negative to reverse the current.

The behavior of aluminium against iron in dilute hydrochloric acid in the cell described above when the terminals are connected through a voltmeter is entirely different. In this case aluminium is positive to iron throughout. When the aluminium and iron electrodes are dipped into the dilute hydrochloric acid, at the outset there is a relatively high reading which gradually drops until maximum polarization is reached after which the needle remains steady. Lifting the iron electrode into the air for a short time increases the voltage reading temporarily, but it soon drops back to the former reading. Lifting the aluminium pole into the air does not change the reading at all. With aluminium and iron electrodes in dilute hydrochloric acid it is not possible to produce a reversal of current as in sulphuric and nitric acids and evidently there is no oxygen or oxide influence at work, or, to state the matter in better form, the oxygen influence is overcome and dominated by the halogen influence. This corresponds to Byers' results on the passivity of iron and other metals since he found that passivity could not be induced in the presence of halogen acids or compounds. The results, therefore, indicate that reversal of the

normal direction of current in oxygen acids is closely connected with passivity phenomena and that the active agent in both classes of phenomena is oxygen.

### THE OXYGEN INFLUENCE: OXIDIZING AGENTS, ACIDS, SALTS, CHROMIC ACID.

It is seen from the foregoing that the oxygen influence makes itself felt not only when free oxygen is the active substance, but also when oxygen is present in an oxidizing compound. In the case of a metal which is extremely sensitive to the oxygen influence such as aluminium, an oxygen acid which is ordinarily not regarded as an oxidizing acid and which in fact under ordinary circumstances is not capable of oxidizing may impose the oxygen influence on the metal. This is seen in the behavior of aluminium in sulphuric acid. Another point of great interest is that the oxygen influence is not exhibited in the presence of halogens.

In a previous paper[7] the behavior of iron made passive by chromic and nitric acid toward untreated iron is discussed.

While iron made passive by chromic acid exhibits the reversal phenomenon in both dilute sulphuric acid and sodium sulphate solution, nickel on the contrary, at first negative to untreated nickel, soon goes to zero potential and remains there, showing no tendency to reverse.

When copper is immersed in chromic acid solution it dissolves rather rapidly. A copper strip treated in this way is at first transiently negative to clean untreated copper, but soon becomes electrically neutral to the untreated electrode and remains so.

Lead exhibits quite another behavior. When dipped into chromic acid solution it becomes covered with a yellow layer (consisting of chromate) and such a coated strip is definitely and steadily negative to untreated lead, showing a very constant potential difference. However, the coated strip of lead is still positive to copper. In fuming nitric acid lead does not dissolve but behaves as iron does and many other metals. A strip of lead after immersion in fuming nitric acid is slightly negative to untreated lead, but the potential soon goes to zero.

[7] Experiments on the Corrosion of Iron and Steel. Proc. Am. Inst. Chem. Eng. (1920), Montreal Meeting.

### THE OXYGEN INFLUENCE: CAST IRON, NICKEL, LEAD.

The oxygen influence as shown in the simple cell affects different metals very differently. Cast iron, for example, is less susceptible to the oxygen influence than pure rolled metal, and this accords with the behavior of cast iron in non-oxidizing and oxidizing acids, the rates of solution in the two being of approximately the same order. Nickel is very susceptible to the oxygen influence, and if one of two clean electrodes in dilute sulphuric acid is raised momentarily into the air, on reimmersion that one becomes electro-negative to the one remaining submerged. If the other one is raised into the air it becomes electro-negative and this can be repeated any number of times. On the other hand, lead is not very susceptible to the oxygen influence as shown by this method of experimentation. Zinc and tin are slightly susceptible; but some effect is shown by all the metals except the more noble ones. In the case of platinum the effect is negligible. As a rule, the more noble the metal the less the oxygen influence, but there are some important exceptions, and the order of the electrochemical series is no more maintained in this instance than in the general cases of corrosion and solution in acids.

### RATES OF SOLUTION IN ACIDS.

It is questionable whether results of experiments on the rates of solution of metals in acids can be exactly duplicated in succeeding experiments, and, therefore, it is advisable whenever possible to compare samples directly in the same acid and the same bath. If even approximately agreeing results are to be obtained, a very careful procedure must be followed and the size of the plates, the preparation of the surface, the strength and temperature of the acid must be strictly regulated.

1. The plates used should be of sufficient size and of suitable dimensions, not less than 4 x 4 inches (10 x 10 cm.) square and preferably 6 x 6 inches (15 x 15 cm.). Rolled plates should be No. 16 gauge, and cast plates ⅛ inch (0.3 cm.) thick or as close to this dimension as possible.

2. Surfaces should be clean and in comparable condition. Sandblasting is not recommended, except for preliminary treatment. Grinding or rubbing with emery to a smooth but not polished surface is probably the best procedure.

3. The acid concentration should be maintained constant either by using a large volume, operating for a short time only (consistent with accurate results) or by circulating the acid through the dissolving vessel from a large reservoir.

4. The temperature of the acid should be maintained constant by the use of a thermostat, by the use of large volume or by circulating the acid through an external cooling device.

5. The length of time for the different classes of metals should be the same so far as possible and (a) long enough to obtain significant losses and establish definite surface conditions, (b) not so long that seriously interfering products collect on the surface, unless (c) a further separate test is desired to ascertain the effect of these substances.

Even when all these conditions are observed, duplicate results may not be identical, but they will be approximately the same, and certainly of the same order.

### COMPARISON OF CORROSION AND ACID SOLUTION IN TESTS.

In the past the principal difficulty seems to have been due to the comparison of unlike things or unlike conditions when an attempt was made to correlate acid solution tests and corrosion tests, for example, when the attempt was made to determine the corrodibility of a sample of iron or steel by the rate of solution in a non-oxidizing acid. Such a comparison might be made for conditions of corrosion wherein little or no oxygen was present. It is now well known that under such conditions corrosion proceeds with extreme slowness and that, in fact, under most conditions for any given kind of iron or steel the amount of corrosion, other things being equal, is proportional to the oxygen available. This rule cannot be considered absolute under actual conditions inasmuch as oxygen, the accelerator of corrosion, also produces adherent rust which may, under some conditions, become a protector against corrosion.

Acid solution tests in non-oxidizing acids can be compared only with corrosion tests under non-oxidizing or practically non-oxidizing conditions; but since water in the presence of which corrosion takes place has such a low concentration of hydrogen ions, whereas the strong acids have such a relatively high concentration, the time factor becomes of the greatest importance, and whereas

grey cast iron, for example, dissolves at a rapid rate in normal sulphuric or hydrochloric acid, it dissolves at a negligible rate in water in the absence of air, although theoretically it should dissolve or corrrode in water much faster than purer irons.

A comparison of the results of corrosion tests under oxidizing conditions cannot be made with acid solution tests unless the acids used are oxidizing acids, which at the same time do not at the concentration employed develop passivity phenomena, or not to a greater extent than does atmospheric oxygen. It is possible that a method might be developed of acting upon metals by non-oxidizing acids in the presence of an abundant air supply and that such tests might be comparable with corrosion tests under oxidizing conditions. While some results obtained indicate a slight acceleration when air is bubbled through normal acid in which an experimental plate was immersed, conclusive results have not been obtained.

What has been said of non-oxidizing acids and oxidizing acids applies also to other conditions. If halogens are present in one set of experiments, they should be present in the other. The influence of cathodes and of the products of corrosion should be brought into play in one case as well as in the other. In brief, it is only by making the two sets of experiments strictly comparable that comparable results can be obtained and the peculiar behavior of each metal must be known before an accurate interpretation of the results can be looked for.

## DISSOLVING OF METALS BY ACIDS IN THE PRESENCE OF CATALYSTS.

Following the suggestion made previously that the solution of metals in acids and corrosion may be considered as actions which tend to go forward in the absence of disturbing conditions at rates proportional to solution pressure and the concentration of hydrogen ions, it was thought that one of the first steps necessary to gain a further knowledge of the behavior of different metals undergoing solution or corrosion would be their behavior in acids with various metallic catalysts. The impurities in iron and steel which influence their rate of solution or corrosion are sometimes of a metallic nature, sometimes non-metallic. The metals as catalyzers are more easily studied than the non-metals, but following a study of the metals as catalyzers, the study of the non-metals

such as silicon, graphite, sulphur (sulphides), arsenic, etc., should be taken up. With these studies in hand and a working knowledge of the hydrogen and oxygen influences on the metals and the various catalysts, the main factors for the correlation of solution and corrosion of metals would be available.

## SOLUTION IN SULPHURIC ACID WITH AND WITHOUT PLATINUM CATALYZER.

Experiments were made on the rate of solution of four kinds of iron, rolled copper and aluminium in normal sulphuric acid at 16° C. with and without the addition of a small amount of platinum. Table I shows the results obtained. The plates used measured 6 x 6 inches (15 x 15 cm.) square and were No. 16 gauge with the exception of the cast metal plates, which were ⅛ inch (3 mm.) thick.

TABLE I.

Solution of iron, copper and aluminium in normal sulphuric acid alone and with the addition of a small amount of platinum salt (0.5 g. Pt in 38 liters). Temperature 16°C.

| | Loss in Grams | | | |
|---|---|---|---|---|
| | $H_2SO_4$ | | $H_2SO_4 + Pt$ | |
| | Loss, 5-hour Period | Average Loss per Hour | Loss, 4-hour Period | Average Loss per Hour |
| Copper bearing iron.. | 0.6 | 0.12 | 19.6 | 4.9 |
| Open hearth pure iron | 3.8 | 0.76 | 61.8 | 15.5 |
| Grey cast iron..... | 44.3* | 8.9* | 38.3* | 9.6* |
| Semi-steel ..... | 44.3* | 8.9* | 38.4* | 9.6* |
| | 8-hr. Period | | | |
| Copper ..... | 0.4 | 0.05 | 0 | 0 |
| Aluminium ..... | 0.12 | 0.02 | 0.3 | 0.04 |

* Weight after sand-blasting to remove adherent layer of graphite, etc.

In the case of the cast metals it will be noticed that losses are given before and after sand blasting for the reason that after the action of the acid there remains on the cast metal surfaces a layer consisting of graphitic carbon, silicides and other impurities disengaged by the action of the acid. No correction has been made for the slight difference in thickness of the cast metals.

It will be seen from the table that the copper bearing iron dis-

solves in normal sulphuric acid at the low rate of 0.12 g. per hour, which is accelerated moderately in the presence of platinum to 4.9 g. The open hearth pure iron dissolves in sulphuric acid alone at the rate of 0.76 g. per hour, and this rate is accelerated to a considerable extent by the addition of platinum, namely, to 15.5 g. per hour. The grey cast iron and the semi-steel dissolve at the same rate in sulphuric acid, 8.9 g. per hour and this rate is catalyzed to a very slight extent by the addition of platinum. As stated before, the effect of poisons was not eliminated.

Copper dissolves in the sulphuric acid alone at an extremely low rate, 0.05 g. per hour being the amount lost, and in the presence of platinum there is no loss of metal whatever.

### SOLUTION IN HYDROCHLORIC ACID WITH AND WITHOUT PLATINUM CATALYZER.

The results in this instance are similar in general to those in sulphuric acid, with the very prominent exception of aluminium. The copper bearing iron and the open hearth pure iron dissolve at somewhat lower rates in hydrochloric than in sulphuric acid, and this may or may not have been due to some condition of the experiment, such as inexact control of the temperatures. The grey cast iron shows a relatively similar loss in hydrochloric acid without platinum and in the presence of platinum. The semi-steel behaves as might be expected. The copper shows the same peculiarity as in sulphuric acid, namely, the slightly lower rate with platinum. The aluminium dissolved at a faster rate in the acid and was enormously catalyzed by the platinum, so that it became difficult to control the temperature, and the figure obtained can only be considered approximate. The difference in the behavior of aluminium in sulphuric and hydrochloric acid is remarkable and may be explained by the difference between the oxygen and the halogen influence. See Table II.

### SOLUTION IN NITRIC ACID WITH AND WITHOUT PLATINUM CATALYZER.

Table III shows the rates of solution in normal nitric acid, and here the oxygen influence is paramount. The pure irons, that is, the copper-bearing iron and the open-hearth pure iron, dissolve at

## Table II.

Solution of iron, copper and aluminium in normal hydrochloric acid alone and with the addition of a small amount of platinum salt (0.5 g. Pt in 38 liters). Temperature 16°C.

| | Loss in Grams | | | |
|---|---|---|---|---|
| | HCl | | HCl + Pt | |
| | Loss, 5-hour Period | Average Loss per Hour | Loss, 5-hour Period | Average Loss per Hour |
| Copper bearing iron.. | 0.1 | 0.02 | 16.0 | 3.2 |
| Open hearth pure iron | 0.6 | 0.12 | 52.6 | 10.5 |
| Grey cast iron..... | 43.5 | 8.7 | 44.0 | 8.8 |
| | 58.3* | 11.7* | 54.0* | 10.8* |
| Semi-steel ..... | 26.8 | 5.3 | 44.4 | 8.9 |
| | 44.5* | 8.9* | 52.7* | 10.5* |
| | 3 Hours | | | |
| Copper ..... | 0.3 | 0.1 | 0 | 0 |
| Aluminium ..... | 10.3 | 3.4 | 11.2 | 3.7 |

* Weight after sand-blasting to remove adherent layer of graphite, etc.

## Table III.

Solution of iron, copper and aluminium in normal nitric acid alone and with the addition of a small amount of platinum salt (0.5 g. Pt in 38 liters). Temperature 16°C.

| | Loss in Grams | | | | |
|---|---|---|---|---|---|
| | $HNO_3$ | | | $HNO_3$ + Pt | |
| | Loss, 1-hour Period | Loss, 8-hour Period | Average Loss per Hour | Loss, 1½-hour Period | Average Loss per Hour |
| Copper bearing iron..... | 35.4 | ..... | 35.4 | 47.8 | 31.8 |
| Open hearth pure iron... | 34.2 | ..... | 34.2 | 49.9 | 33.2 |
| Grey cast iron ..... | 22.2 | ..... | 22.2 | 30.7 | 20.5 |
| | 25.3* | ..... | 25.3* | 35.4* | 23.6* |
| Semi-steel ..... | 22.0 | ..... | 22.0 | 32.1 | 21.4 |
| | 25.7* | ..... | 25.7* | 37.6* | 25.0* |
| | | | | 5 Hours | |
| Copper ..... | ..... | 0.1 | 0.01 | 0 | 0 |
| Aluminium ..... | ..... | 0.1 | 0.01 | 0.3 | 0.06 |

* Weight after sand-blasting to remove adherent layer of graphite, etc.

very rapid rates in the nitric acid alone and this action is not accelerated by platinum. In fact, the actual figures obtained in the presence of platinum are slightly less than in the acid without the

platinum. This difference, however, may have been due to slight differences in the conditions of the experiments, but the conclusion is warranted that platinum does not accelerate the solution of these irons in nitric acid. The same holds for the two cast metals, which dissolve at a lower rate than the purer metals in the nitric acid, and platinum does not exert an accelerating effect. The higher rate of solution of the purer irons over that of the cast iron is a matter of great interest and importance. Copper dissolves to a very slight extent only in the normal nitric acid and to a slightly less extent in the presence of platinum. The results for

TABLE IV.

Solution of iron, copper and aluminium in normal acids alone and with the addition of a small amount of platinum salt (0.5 g. Pt in 38 liters). Temperature 16°C.

| | Loss in Grams per Hour | | | | | |
|---|---|---|---|---|---|---|
| | HCl | HCl + Pt | $H_2SO_4$ | $H_2SO_4$ + Pt | $HNO_3$ | $HNO_3$ + Pt |
| Copper bearing iron.... | 0.02 | 3.2 | 0.12 | 4.9 | 35.4 | 31.8 |
| Open hearth pure iron.. | 0.12 | 10.5 | 0.76 | 15.5 | 34.2 | 33.2 |
| Grey cast iron..... | 8.7 | 8.8 | 8.9* | 9.6* | 22.2 | 20.5 |
| | 11.7* | 10.8* | | | 25.3* | 23.6* |
| Semi-steel ..... | 5.3 | 8.9 | 8.9* | 9.6* | 22.0 | 21.4 |
| | 8.9* | 10.5* | | | 25.7* | 25.0* |
| Copper ..... | 0.1 | 0 | 0.05 | 0 | 0.01 | 0 |
| Aluminium ..... | 3.4 | 3.7 | 0.02 | 0.04 | 0.01 | 0.06 |

* Weight after sand-blasting to remove adherent layer of graphite, etc.

aluminium show no appreciable catalytic effect of the platinum on the rate of solution, which is extremely low in both cases. In general, then, platinum does not catalyze the action of metals on nitric acid unless possibly in a negative manner. Hydrogen released by electrolytic action is presumably oxidized promptly to water and removed. The cathode influence, therefore, is of no avail in promoting solution.

## THE PURER IRONS.

As the tables show (the results are given in condensed form in Table IV) the purer irons, such as open hearth and copper bearing, dissolve at a low rate in the non-oxidizing acids, hydrochloric

and sulphuric acid (a reservation must be made for sulphuric acid since, as is suggested below, it apparently exerts an oxidizing influence in the case of aluminium). Solution is accelerated by the presence of platinum in the case of both acids, but the rate of solution is much lower for copper-bearing metal than for the purer open-hearth iron.

In nitric acid the rates for copper-bearing and open-hearth pure iron are practically equalized and extremely high and platinum exerts no accelerating effect. The platinum in this instance was added to the solution in the form of nitrate produced by evaporating platinum chloride several times with nitric acid. It might be thought that in the presence of nitric acid, platinum is not deposited upon the metal surface and hence that no catalytic action resulted. However, in the case of copper and aluminium the plates used were taken from previous experiments in sulphuric and hydrochloric acid, and the plates, therefore, had platinum already plated out on them from the previous experiments.

## THE CAST METALS.

The grey cast iron and semi-steel[6] form an interesting series compared to the purer irons. They dissolve much more readily in hydrochloric and sulphuric acid (the rate is of the same order in both acids) and the action is not greatly catalyzed by platinum, although there are indications of a moderate accelerating action. In nitric acid the rate is approximately doubled over that in the non-oxidizing acids plus platinum, and platinum exerts no accelerating influence.

## COPPER.

This metal goes into solution in all three acids at a very low rate, and probably would not dissolve at all in the absence of oxygen. Platinum possibly exerts a negative catalytic effect, a matter which requires further investigation. The copper plate used was darkened by the deposition of platinum upon it, and the same plate was used for all three experiments, so that there was a deposit of platinum on the plate in the nitric acid experiment.

[8] For analyses and descriptions of all the metals used the reader is referred to the paper "Experiments on the Corrosion of Iron and Steel." Proc. Am. Inst. Chem. Eng. (1920).

### ALUMINIUM.

Aluminium behaves in a peculiar manner in all acid and corrosion experiments. In hydrochloric acid alone it dissolves at a fair rate, and this rate is enormously catalyzed by platinum. On the other hand, in sulphuric acid the platinum exerts no catalytic effect whatever in a positive direction, although it may catalyze negatively. Aluminium also fails to dissolve to any appreciable extent in normal nitric acid and platinum fails to catalyze the action. The behavior of aluminium is probably best explained by the oxygen influence and apparently sulphuric acid is capable either of exerting an oxidizing influence on the aluminium surface or is unable to overcome the oxidizing effect of atmospheric oxygen, whereas hydrochloric acid, well-known to be, like all halogen acids, an adverse influence in passivity phenomena, is readily attacked by the aluminium. The oxide layer or the passive layer on the metal surface apparently cannot exist in the presence of the halogen acid. Nitric acid also acts in such a way as to maintain the oxide or passive layer on the aluminium, thus preventing solution.

### THE ACTION OF PLATINUM.

If it is true that platinum when it catalyzes the action of iron on non-oxidizing acids merely supplies cathodes which allow of the easy disengagement of hydrogen and thus prevent polarization, catalysis of this sort admits of easy explanation. In cast iron apparently this function is performed by graphitic carbon, which is present in considerable volume. In nitric acid there is no opportunity for polarization, hence platinum does not catalyze. On the other hand, there is an opportunity for the oxygen influence to show itself and to inhibit action in the case of metals easily influenced to produce an oxygen protective layer.

### SOLUTION IN ACIDS WITH SILVER AS CATALYZER: TABLE V.

The silver was introduced in the form of chloride, sulphate and nitrate in the respective acids, the concentration being in each case 10 grams metallic silver in 100 liters of acid. No noticeable effect was produced on any of the metals tried in hydrochloric or sulphuric acids. In nitric acid the silver catalyzed the solution of the copper-bearing and open-hearth pure iron negatively, re-

ducing the rate about 50 percent. On the other hand, the rates for the grey cast iron and semi-steel were accelerated, but only moderately.

TABLE V.

Solution of iron, copper and aluminum in normal acids in the presence of small amount of silver. 10 g. of silver in 100 liters of acid. Temperature 16°C.

| | *N*/1 Hydrochloric Acid | | *N*/1 Sulphuric Acid | | *N*/1 Nitric Acid | |
|---|---|---|---|---|---|---|
| | Total Loss for 2-hour Period | Av. Loss per Hour | Total Loss for 3-hour Period | Av. Loss per Hour | Total Loss for 1-hour Period | Av. Loss per Hour |
| CC Copper bearing iron ..... | 0.2 | 0.1 | 0.0 | 0.0 | 15.5 | 15.5 |
| KK Open hearth pure iron .... | 0.1 | 0.05 | 0.2 | 0.06 | 15.9 | 15.9 |
| | 1-hr. Period | | 2-hr. Period | | | |
| FF Grey cast iron.. | 7.3<br>10.8* | 7.3<br>10.8* | 14.5<br>19.1* | 7.25<br>9.55* | 30.8<br>36.1* | 30.8<br>36.1* |
| | 1-hr. Period | | 2-hr. Period | | | |
| GG Semi-steel ..... | 7.1<br>10.2* | 7.1<br>10.2* | 14.0<br>20.1* | 7.0<br>10.05* | 33.9<br>38.5* | 33.9<br>38.5* |
| | | | | | 4-hr. Period | |
| Copper ..... | 0.0 | 0.0 | 0.1 | 0.03 | 0.6 | 0.15 |
| | 1-hr. Period | | | | | |
| Aluminium ..... | 0.6 | 0.6 | 0.3 | 0.1 | 0.8 | 0.20 |

* Weight after sand-blasting to remove adherent layer of graphite, etc.

SOLUTION IN ACIDS WITH COPPER AS CATALYZER: TABLE VI.

The copper was added in the form of chloride, sulphate and nitrate to the respective acids, 10 grams metallic copper being present per 100 liters of acid. In hydrochloric and sulphuric acid none of the iron plates showed any appreciable change of rate, although the cast metals in hydrochloric acid showed somewhat greater losses than those in sulphuric acid. Still, in both cases the rates were of the same order and of about the same order as when no catalyzer was used. Aluminium showed a somewhat increased rate in sulphuric acid, but the rate was not very large, slightly greater than 1 gram per hour.

On the other hand, copper appears to exert its greatest influence in nitric acid. As has previously been shown[9] the comparatively

[9] Rates of Solution of Iron and Steel in Non-oxidizing and Oxidizing Acids. Proc. Am. Inst. Chem. Eng. (1920).

pure rolled metals dissolve in normal nitric acid at 16° C. on the average about twice as rapidly as do the grey cast irons and the semi-steels. When a small amount of copper is added to the normal nitric acid—in this instance the equivalent of 10 grams of metallic copper per 100 liters of acid—it is found that the rate of solution for the purer metals is catalyzed negatively so that in the presence of copper the rate is lowered to about two-thirds the rate without copper. In the case of the cast irons and semi-steels on the contrary the rate is catalyzed positively and is on the average approximately doubled.

TABLE VI.

Solution of iron, copper and aluminium in various acids with a small amount of copper present. 10 g. of copper in 100 liters. Temperature 16°C.

| | Hydrochloric Acid | | Sulphuric Acid | | Nitric Acid | |
|---|---|---|---|---|---|---|
| | Total Loss, 2-hour Period | Av. Loss per Hour | Total Loss, 5-hour Period | Av. Loss per Hour | Total Loss, 2-hour Period | Av. Loss per Hour |
| Copper bearing iron. | 0.2 | 0.1 | 0.1 | 0.02 | 50.3 | 25.1 |
| Open hearth pure iron | 0.8 | 0.4 | 0.3 | 0.06 | 45.4 | 22.7 |
| Grey cast iron ..... | 16.8<br>21.5* | 8.4<br>10.7* | 31.8<br>42.3* | 6.3<br>8.4* | 51.1<br>55.0* | 25.5<br>27.5* |
| Semi-steel ..... | 15.6<br>21.1* | 7.8<br>10.5* | 31.8<br>41.1* | 6.3<br>8.2* | 53.5<br>56.7* | 26.7<br>28.3* |
| | | | | | 4 hr. | |
| Copper ..... | 0 | 0 | 0 | 0 | 0 | 0 |
| | 1 hr. | | Gain in Weight | Gain per Hour | 4 hr. | |
| Aluminium ..... | 0.9 | 0.9 | 5.9 | 1.2 | 1.2 | 0.3 |

* Weight after sand-blasting to remove adherent layer of graphite, etc.

The copper deposits on the surface of the dissolving metals even from nitric acid. The deposited copper acting as a catalyst is evidently not poisoned by the sulphides and other impurities arising from the cast metals. According to the older views of the electrolytic theory, copper in contact with pure iron ought to catalyze positively in non-oxidizing acids, but this is not the case. Its effect on the purer metals is negligible, nor does it affect the cast metals appreciably in non-oxidizing acids. Its influence seems to

be felt only in nitric acid of those studied, whether in a positive or negative direction, and this influence appears to negative the peculiar action of nitric acid on the metals as contrasted with the non-oxidizing acids. In the case of those metals where nitric acid causes an increased rate of solution, the presence of copper diminishes this action, and in cases where nitric acid lowers the rate below that of the non-oxidizing acids, the presence of copper increases the rate.

TABLE VII

Solution of iron, copper and aluminium in normal nitric acid with and without metallic copper.

| | *N*/1 Nitric Acid. Temp. 16° C. | |
|---|---|---|
| | Without Copper. Av. Loss per Hour | 10 gm. Metallic Copper per 100 liters of Acid. Av. Loss per Hour |
| CC Copper bearing iron | 30.0 | 22.5 |
| KK Open hearth pure iron | 34.7 | 18.9 |
| FF Grey cast iron | 10.7<br>13.5* | 31.0<br>34.8* |
| GG Semi-steel | 8.9<br>11.7* | 26.1<br>32.3* |
| Copper | 0.0 | 0.0 |
| Aluminium | 0.1 | 0.0 |

* Weight after sand-blasting to remove adherent layer of graphite, etc.

The net result of this is to bring the rates for the relatively pure irons in nitric acid below those of the cast metals. This point is brought out again in the figures shown in Table VII, which represent a second experiment with and without copper in nitric acid.

Table VIII gives the results on two different samples of four kinds of iron, duplicates (1 and 2) being run on separate plates. These figures, generally speaking, show a drop to two-thirds in the case of the rolled metals in the presence of copper and an increase of approximately 100 percent in the case of the cast metals.

The reduced rates for the purer metals in nitric acid in the presence of copper as catalyzer are doubly interesting on account

of the claims that have been made for the corrosion-resisting qualities of copper-bearing iron. It will be noticed that the results show a reduction in rate, however, both for the copper-bearing and

TABLE VIII.

Solution of various irons in $N/1$ Nitric Acid without and with small amount of copper. 10 g. metallic copper per 100 liters of acid.

| | Temperature 16° C. | | | |
|---|---|---|---|---|
| | No. | $N/1$ Nitric Acid. Loss for 1 Hour | No. | $N/1$ Nitric Acid and Copper. Loss for 1 Hour |
| AA Copper bearing iron..... | 1 | 32.1 | 1 | 21.5 |
| | 2 | 30.6 | 2 | 22.9 |
| CC Copper bearing iron..... | 1 | 29.8 | 1 | 22.5 |
| | 2 | 31.9 | 2 | 20.7 |
| BB Open hearth pure iron..... | 1 | 31.8 | 1 | 19.0 |
| | 2 | 31.3 | 2 | 22.4 |
| KK Open hearth pure iron..... | 1 | 34.7 | 1 | 18.9 |
| | 2 | 31.1 | 2 | 21.8 |
| FF Grey cast iron..... | 1 | 10.7 | 1 | 31.0 |
| | | 13.5* | | 34.8* |
| | 2 | 11.6 | 2 | 31.2 |
| | | 16.0* | | 35.6* |
| SS Grey cast iron ..... | 1 | 8.2 | 1 | 17.4 |
| | | 12.2* | | 25.3* |
| | 2 | 10.2 | 2 | 28.9 |
| | | 16.1* | | 34.4* |
| GG Semi-steel ..... | 1 | 8.9 | 1 | 26.1 |
| | | 11.7* | | 32.3* |
| | 2 | 12.1 | 2 | 30.7 |
| | | 15.5* | | 34.7* |
| RR Semi-steel ..... | 1 | 14.9 | 1 | 28.6 |
| | | 18.7* | | 32.1* |
| | 2 | 10.0 | 2 | 20.0 |
| | | 14.9* | | 28.4* |

* Weight after sand-blasting to remove adherent layer of graphite, etc.

the open-hearth pure iron, indicating that if copper has a tendency to decrease the rate under oxidizing conditions that the amount commonly added to copper-bearing mild steel does not produce the maximum effect.

SOLUTION IN ACIDS WITH NICKEL AS CATALYZER: TABLE IX.

Nickel when added as chloride, sulphate and nitrate to the respective acids in concentration equivalent to 10 grams of metallic nickel per 100 liters, exerted practically no effect on the rates of solution of any of the metals in any of the acids with the exception of aluminium in hydrochloric acid. This rate was catalyzed from its normal rate of something less than 1 gram per hour in pure acid to 16 grams per hour. In sulphuric acid the rate was catalyzed only slightly.

TABLE IX.

Solution of iron, aluminium and copper in various acids with a small amount of nickel present. 10 g. of metallic Ni per 100 liters. Temperature 16°C.

| | Hydrochloric Acid | | Nitric Acid | | Sulphuric Acid | |
|---|---|---|---|---|---|---|
| | Total Loss, 2-hour Period | Av. Loss per Hour | Total Loss, 5-hour Period | Av. Loss per Hour | Total Loss, 1-hour Period | Av. Loss per Hour |
| Copper bearing iron ..... | 0.4 | 0.2 | 0.3 | 0.06 | 32.1 | 32.1 |
| Open hearth pure iron.... | 0.6 | 0.3 | 1.3 | 0.26 | 28.7 | 28.7 |
| Grey cast iron. | 16.2<br>23.1* | 8.1<br>11.6* | 33.1<br>43.3* | 6.62<br>8.66* | 11.4<br>15.8* | 11.4<br>15.8* |
| Semi-steel .... | 15.0<br>23.3* | 7.5<br>11.7* | 31.8<br>43.5* | 6.36<br>8.70 | 12.0<br>16.8* | 12.0<br>16.8* |
| Copper ..... | 0.4 | 0.2 | 4 hr.<br>0 | 0 | 5 hr.<br>0.9 | 0.18 |
| Aluminium ... | 30 min.<br>8.0 | 16.0 | 1.4 | 0.35 | 0.8 | 0.16 |

* Weight after sand-blasting to remove adherent layer of graphite, etc.

BEHAVIOR OF AN ORGANIC CATALYST, FORMALDEHYDE.

In Table X are set forth losses from copper-bearing iron, open-hearth pure iron, grey cast iron and semi-steel in normal sulphuric, hydrochloric and nitric acids plus 1 percent actual formaldehyde in each case. In the non-oxidizing acids the purer rolled metals show little change of rate, but the solution of the cast metals is strongly catalyzed in a negative direction to about one-third of their usual rate in normal acids. In nitric acid the rates of the pure metals, on the other hand, are catalyzed negatively to about

one-half their usual amounts, while those for the cast metals are accelerated by about 50 per cent. In general, it may be said of these results that they emphasize once more the fact that the behavior with or without catalyzers in non-oxidizing acids is different from that in oxidizing, and again that the behavior of the purer rolled metals with or without catalyzers is different from that of the cast metals. The results with formaldehyde are inserted at this point, largely for the purpose of contrasting them with the results obtained by the use of metallic catalyzers. It is difficult to see that there can be any similarity in mode of action

TABLE X.

Rates of solution of 6 x 6 inch (15 x 15 cm.) iron plates in normal acids at 16°C. alone and with the addition of formaldehyde. 1 percent actual formaldehyde present. Losses in grams per hour.

| | $H_2SO_4$ | $H_2SO_4$ + Formaldehyde | HCl | HCl + Formaldehyde | $HNO_3$ | $NHO_3$ + Formaldehyde |
|---|---|---|---|---|---|---|
| CC Copper bearing iron ..... | 0.3 | 0.2 | 0.2 | 0.15 | 31. | 17.0 |
| KK Open hearth pure iron..... | 0.3 | 0.2 | 0.2 | 0.0 | 30. | 17.2 |
| FF Grey cast iron.. | 6.9<br>10.4* | 1.3<br>2.7* | 7.5<br>9.5* | 1.0<br>3.1* | 16.0<br>22.0* | 29.0<br>36.0* |
| GG Semi-steel ..... | 7.6<br>11.0* | 1.0<br>2.5* | 7.8<br>11.1* | 1.3<br>3.7* | 16.2<br>23.1* | 28.0<br>35.0* |

* Weight after sand-blasting to remove adherent layer of graphite, etc.

between a metallic catalyzer and a soluble organic substance such as formaldehyde, and yet in the case of copper, we find the rates for the purer irons catalyzed negatively and the rates for the cast metals catalyzed positively as in the case of formaldehyde. Naturally, the negative and positive accelerations are not the same for copper and for formaldehyde, because, until the exact mode of action for each substance is known, comparable amounts cannot be added.

## SUMMARY OF CATALYZER EXPERIMENTS.

Table XI summarizes the action of the various catalysts tried on the irons, the copper and the aluminium. Numerous instances of similar action are apparent. In nitric acid, platinum, silver,

copper and formaldehyde catalyze the rate of solution of the purer rolled metals negatively, while nickel shows no action. Platinum catalyzes these metals in sulphuric and hydrochloric acid, and the pure open-hearth more strongly than the copper-bearing. Where zero action is reported, the conclusion is not necessarily warranted that there was no action. It is entirely possible that by the use of

TABLE XI.

*Summary of Catalytic Action of Metals and Formaldehyde.*

| | Copper Bearing Iron | | | Pure Open Hearth Iron | | | Grey Cast Iron | | | Semi Steel | | | Copper | | | Aluminium | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | $H_2SO_4$ | HCl | $HNO_3$ | $H_2SO_4$ | HCl | $HNO_3$ | $H_2SO_4$ | HCl | $HNO_3$ | $H_2SO_4$ | HCl | $HNO_3$ | $H_2SO_4$ | HCl | $HNO_3$ | $H_2SO_4$ | HCl | $HNO_3$ |
| Platinum . . . . | + | + | – | ++ | ++ | – | 0 | 0 | 0 | 0 | 0 | 0 | –? | –? | –? | 0 | +++ | 0 |
| Silver . . . . . | 0 | 0 | – | 0 | 0 | – | 0 | 0 | + | 0 | 0 | + | 0 | 0 | 0 | 0 | 0 | 0 |
| Copper . . . . . | 0 | 0 | – | 0 | 0 | – | 0 | 0 | + | 0 | 0 | + | 0 | 0 | 0 | 0 | 0 | 0 |
| Nickel . . . . . | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | ++ | 0 |
| Formaldehyde | 0 | 0 | – | 0 | 0 | – | – – | – – | ++ | – – | – – | ++ | No data | | | No data | | |

0 Without pronounced influence.
– Catalyzing negatively.
– – Catalyzing more strongly negatively.
+ Catalyzing positively.
+ + Catalyzing more strongly positively.
+ + + Catalyzing still more strongly positively.
? Action doubtful.

larger quantities some catalytic action would be recorded. For example, platinum catalyzes the rolled metals in the non-oxidizing acids positively; at greater concentrations silver and copper might also show this effect.

Platinum produced no effect on the grey cast iron or semi-steel, but this may have been, and probably was, due to the action of catalyzer poisons evolved by the acids from the metals. On the other hand, their rates were catalyzed positively in nitric acid by silver, copper and formaldehyde. In the non-oxidizing acids none

of the catalysts showed activity, with the exception of formaldehyde, which catalyzed the cast metals negatively.

The rate for copper was affected very little by the different catalysts experimented with, but there seemed to be a slight negative tendency in the case of platinum in all three acids. Aluminium was only affected by two of the catalysts, platinum and nickel, and only in hydrochloric acid. No change in rate was observed either in sulphuric or nitric acids. This is probably related in some way to the oxygen influence to which aluminium has already been shown to be extremely sensitive. Platinum and nickel as catalyzers frequently behave in a similar manner, for example, in hydrogenation. From periodic considerations, palladium, too, should show a behavior similar to that of platinum and nickel. It is difficult to harmonize the selective action of platinum and nickel on aluminium in hydrochloric acid with the theory of catalytic action which postulates merely a lower hydrogen overvoltage on the catalytic metal. Apparently there is a reciprocal relationship between the dissolving metal and the acid concerned, or a relationship between all three factors, the dissolving metal, the acid and the catalyst. Neither platinum nor nickel catalyze the solution of aluminium in sulphuric or nitric acids. Even after nitric acid is disposed of on the assumption of its considerable oxygen influence, or the possibility of passivity phenomena, it is difficult to dispose of sulphuric acid in the same way. As ordinarily considered, sulphuric acid is not an oxidizing agent in dilute solution. Apparently in this case we must look to action at the anodes for the explanation. The effect of the anion chlorine $Cl^-$ on the surface of the aluminium must be decidedly different from that of the anion $SO_4^{--}$ and this different relationship is closely connected with the different behavior of these anions in passivity experiments as described by Byers and others, and in the polarity experiments described in an earlier section of this paper. At the corrosion cathodes, substances and conditions are identical. The cathodes, according to theory, would be platinum, and the cations hydrogen.

It is realized that the results reported in this paper are incomplete and of a fragmentary nature. Much remains to be done in extending the list of metals whose rates are being determined, as

well as those used as catalysts. The list of organic catalysts and their effects also require extension. I desire to acknowledge the assistance of Messrs. O. T. Bloom and J. DeRose in the work reported.

## CONCLUSIONS.

1. Inconsistent behavior of the metals in corrosion and acid solution phenomena on the basis of the electrochemical series can only be explained by taking into consideration a number of factors.

2. The principal factors are solution tension, the hydrogen influence, the oxygen influence, the carbon dioxide influence, the cathode influence and the halogen influence.

3. Corrosion and solution of metals in acids may be considered as actions tending to go forward at rates proportional to solution tension and hydrogen ion concentration, but subject to the accelerating and retarding influence of many substances acting as catalyzers.

4. Indications of the sensitiveness of various metals to the hydrogen and oxygen influences can be obtained by their behavior when treated as couples in dilute acids and neutral salts and connected through a sensitive voltmeter or galvanometer.

5. Studying the behavior of metallic catalyzers on the rates of solution of copper-bearing iron, pure open-hearth iron, grey cast iron and semi-steel in normal sulphuric, hydrochloric and nitric acids at 16° C., with several metallic catalyzers, it was found that the rolled metals in general showed a different and opposite behavior to that of the cast metals.

6. Again, the behavior of any of these metals in the non-oxidizing acids, sulphuric and hydrochloric, is as a rule different from the behavior in nitric acid.

7. A similar rule holds for these metals in corrosion and also for solution in the above acids without catalyzers.

8. Although the mode of action of a soluble organic catalyst, such as formaldehyde, is probably different from that of the metallic catalysts, nevertheless, the opposite tendency in the case of different metals in oxidizing and non-oxidizing acids is revealed in this case also.

9. The rates of solution of copper-bearing and pure open-hearth iron in nitric acid are catalyzed negatively by silver, copper and formaldehyde, but the rates of grey cast iron and semi-steel are catalyzed positively by the same three catalysts.

10. No pronounced change of rate is shown by silver, copper or formaldehyde acting as catalysts on copper-bearing or pure open-hearth iron in sulphuric or hydrochloric acid.

11. The same is true for silver and copper when acting on grey cast iron and semi-steel, but in contrast with this behavior, formaldehyde catalyzes these metals strongly in a negative direction in the non-oxidizing acids.

12. Platinum catalyzes the rates of copper-bearing and pure open-hearth iron positively in normal sulphuric and hydrochloric acids, and the action is more pronounced in the case of pure open-hearth iron than in the case of copper-bearing iron.

13. On the contrary, the rates of these metals in normal nitric acid are catalyzed negatively by platinum.

14. The rate for copper is not certainly catalyzed by any of the catalysts used.

15. Nickel as a catalyst produces no effect on any of the metals in any of the acids except aluminium in hydrochloric acid.

16. Aluminium is not affected by any of the catalysts in nitric or sulphuric acid.

17. In normal hydrochloric acid the rate for aluminium is catalyzed strongly by nickel and still more strongly by platinum.

18. A similar action might be expected in sulphuric acid and the lack of action in this case is probably due to the oxygen influence of sulphuric acid on aluminium.

19. Under conditions of corrosion the cast metals are relatively less attacked in the presence of much oxygen than the purer rolled metals.[10] They are also relatively less attacked by an oxidizing acid, such as nitric, than by the non-oxidizing acids, sulphuric and hydrochloric. The catalysts, silver, copper and formaldehyde, all reverse this action and cause the rates of the cast metals in nitric acid to be increased and the rates of the copper-

[10] Experiments on the Corrosion of Iron and Steel, Proc. Inst. Chem. Eng. (1920).

bearing and pure open-hearth irons to be reduced. Platinum, while catalyzing the rolled metals negatively in nitric acid, catalyzes them positively in sulphuric and hydrochloric acid, and is without action on the cast metals in any acid. The failure of platinum to catalyze the cast metals is probably due to the evolution of poisons from the impure metals by the action of the acids.

*A paper presented at the Thirty-eighth General Meeting of the American Electrochemical Society, in Cleveland, September 30, 1920, President Landis in the Chair.*

# PHYSICAL CHARACTERISTICS OF SPECIALIZED REFRACTORIES.[1]

By M. L. Hartmann[2], A. P. Sullivan[3] and D. E. Allen.[3]

## Abstract.

Tests of the electrical resistivities of nine commercially used refractory materials, at temperatures up to 1500° C. Measurements were taken while heating up slowly and while cooling down slowly, the thermocouple measuring temperature being outside the specimens. The amount of temperature lag thus involved was approximately determined. The results are necessarily only approximate, but show the relative resistivities of the different materials and the general order of magnitude of their resistivities up to 1500° C.—[J. W. R.]

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## PART III: ELECTRICAL RESISTIVITY AT HIGH TEMPERATURES.

In the design of electric furnaces it is important to know the electrical resistivity of the refractories used in those portions of the furnace which contain, or come in contact with, the electrodes or resistor material. Practically no data have been published on the resistivities of commercial bricks except those by Stansfield, McLeod and McMahon.[4] These investigators determined the resistivities of silica, chrome, magnesia and fireclay from 600° C. to 1565° C. Hering[5] has compiled from the literature the resistivities of many substances, but these do not include commercial refractories.

The present investigation is concerned with the determination of electrical resistivities of nine commercial refractory bricks up

[1] This paper is the third paper of a series on the physical properties of specialized refractories. Previous installments have been published in these Transactions, Volume 37, 707 and 717. Manuscript received July 22, 1920.

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[4] Trans. Amer. Electrochem. Soc. (1912), 22, 89.

[5] Met. and Chem. Eng. (1915), 13, 23.

to temperatures of 1500° C. The method, in general, is similar to that used by Stansfield, McLeod and McMahon. A large two-core resistance furnace was used for heating the specimens, which were enclosed in a "carbofrax" cylinder about six inches (15 cm.) in diameter.

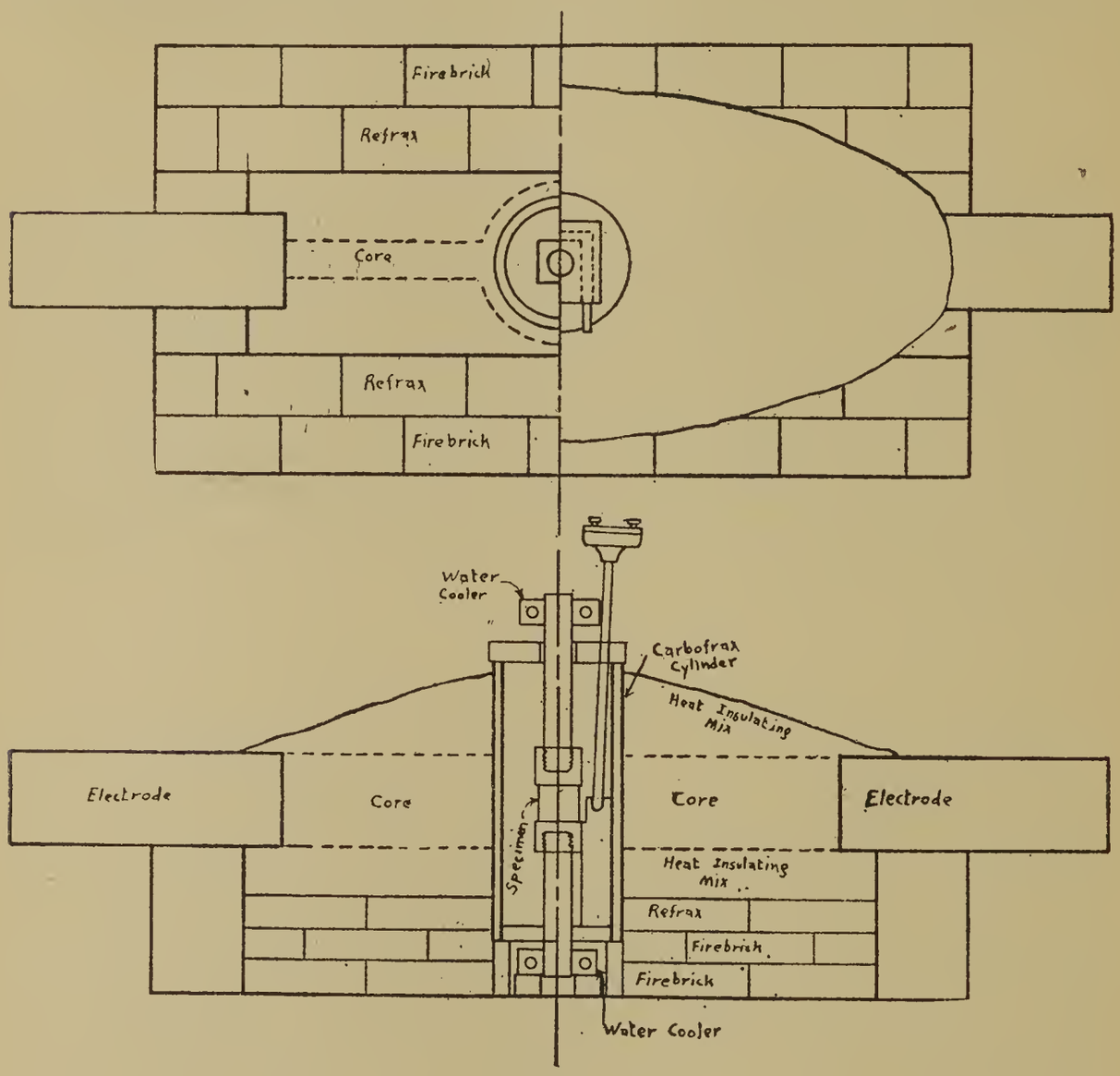


DIAGRAM 1.

The graphite resistor material was placed around this cylinder in such a way as to obtain uniform heating. A two-inch (5 cm.) graphite rod having a water cooler at the bottom extended up into the cylinder, and to the top of this rod a 3½-inch (9 cm.) cubical graphite block was attached (by threaded joint). The specimen was placed on this block, and another graphite block attached to a two-inch graphite rod (as above described) extended down on top of the specimen. This latter contact piece was carefully insulated from the furnace.

The specimens were cut from commercial bricks and ground to 2½-inch (6.4 cm.) cubes. A layer of fine graphite was placed between the graphite contact pieces and the specimen. It was found that a variation in pressure on the top contact piece of from five to one hundred pounds did not affect the contact resistance. In all of the measurements a total of 27 pounds (12.3 kg.) pressure was used.

The heating was controlled at the rate of about 10° C. per minute up to 1000° C. and from three to five degrees per minute from 1000° to 1500° C. The same rate was maintained during cooling.

The temperature was measured by means of a platinum-platinum rhodium thermocouple, in a porcelain protection tube. The couple was so placed in the furnace that the junction was opposite the center of and as close to the specimen as the protection tube would allow. The thermocouple was calibrated at the freezing points of zinc and copper, and the equation[6]

$$\sum_{o}^{t} \epsilon = mt^{n}$$

was assumed to hold over the range of temperature used.

In placing the thermocouple outside the specimen, a higher temperature is measured than the average temperature of the brick. If the thermocouple were placed inside the specimen, a lower temperature would be measured. In order to find the magnitude of this thermal lag, after the resistance of all the bricks had been determined, a hole was drilled through the top graphite contact piece and half way through the specimen of fireclay. A calibrated thermocouple in a porcelain protection tube was thus placed inside the brick and, by taking simultaneous readings inside and outside, the time-temperature lag was determined. A fireclay brick was used because it probably has the lowest thermal conductivity of any of the bricks measured, and would, therefore, show the maximum lag.

The average lag up to 500° C. (heating rate 10° per minute) was 100° C.; from 500° C. to 1000° C. (heating rate 10° per minute) was 65°; from 1000° C. to 1500° C. (heating rate 5° per minute) was 30°. This lag is high at low temperatures, but

[6] Burgess-LeChatelier, "Measurement of High Temperatures," 3d Edition, p. 112.

the actual specific resistances at low temperatures are very great in most specimens, hence this uncertainty in temperature is relatively unimportant. Furthermore, it should be noted that the average temperature of the whole specimen is nearer the outside temperature than the inside. The lag at the high temperatures is not objectionably large when taking into consideration that the thermal conductivities of the materials measured are higher than fireclay, and the lag correspondingly less.

The resistances were measured, at intervals of about 100° C., by means of an "Evershed Bridge Megger" set. In some cases a polarizing effect was noticed and it was impossible to make accurate measurements with the "Megger." This effect occurred

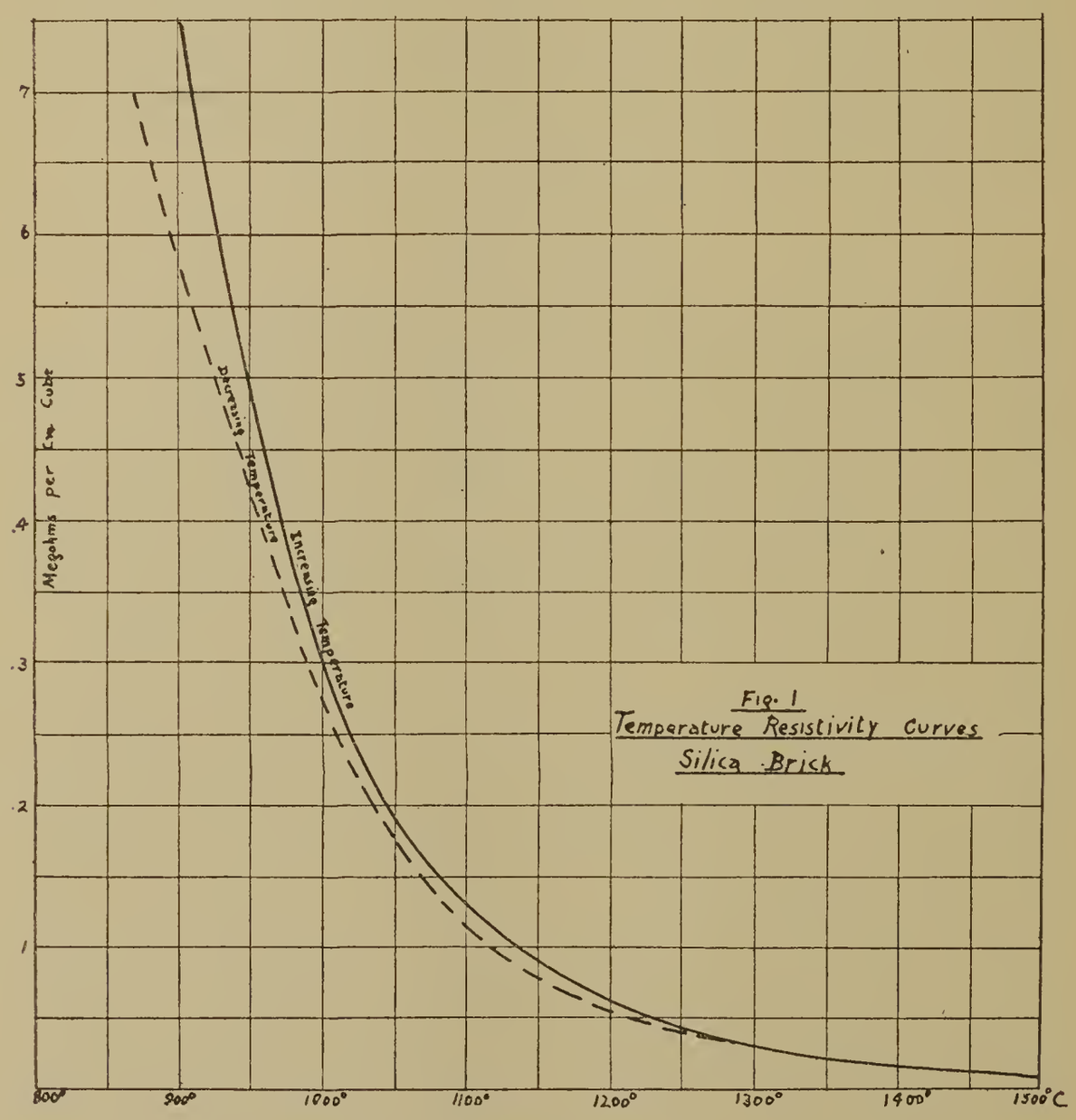


FIG. 1.

principally with "Carbofrax," but was noticed to a lesser degree in other materials. In the case of "Carbofrax," the method of instantaneous readings of impressed voltage and current was employed and the resistance calculated. A direct current of 160–175 volts was used and the maximum deflections of the milli-

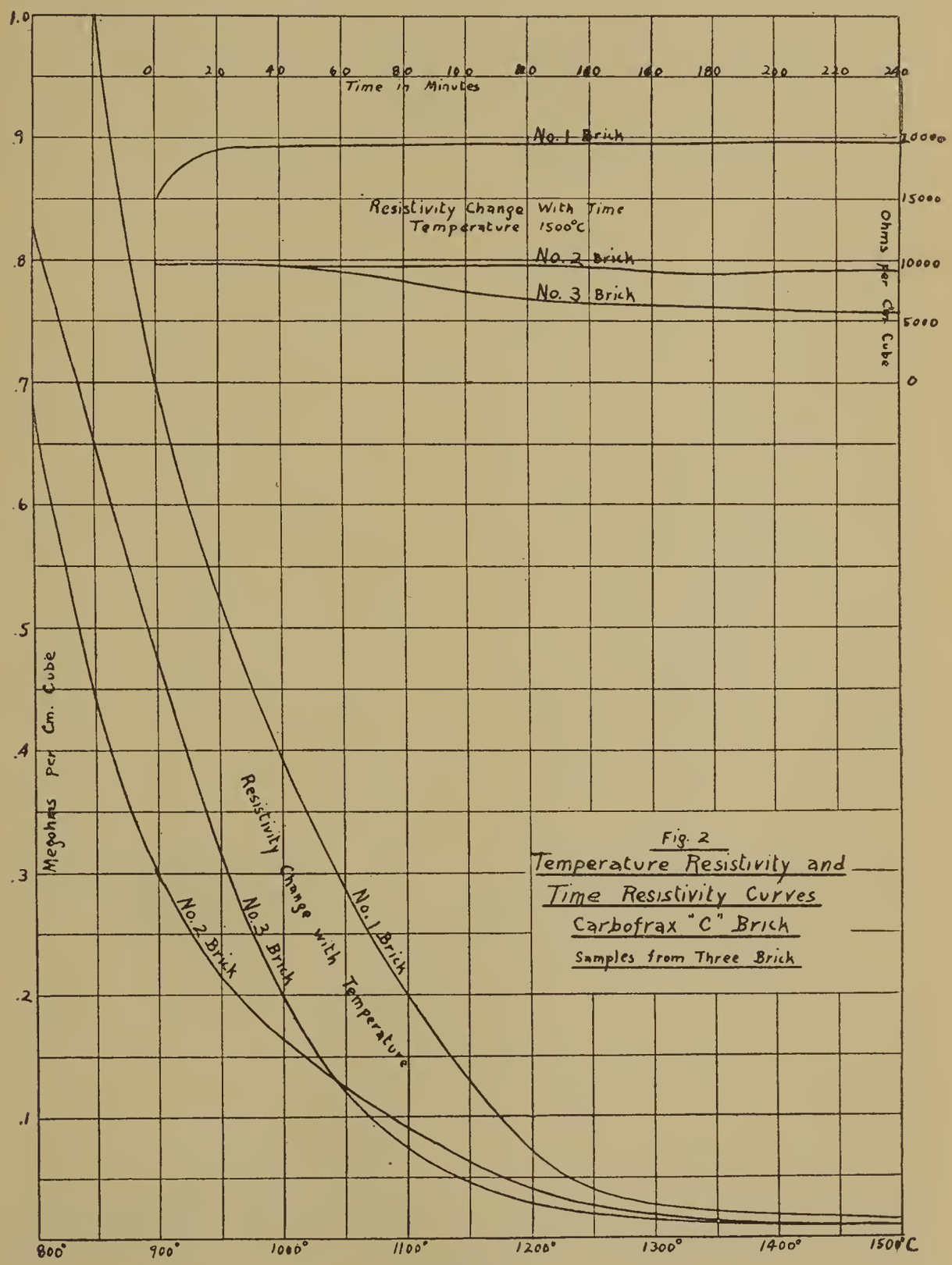


**Fig. 2.**

ammeter upon quick reversals of current were taken as those representing the true resistances.

## *Silica Brick.*

1. Silica bricks (apparent density 1.66) of lime-bond standard make, gave quite regular resistivity curves as shown in Fig. 1. The difference in the position of the increasing and decreasing temperature curves probably represents the lag of the thermocouple temperatures behind the average temperatures of the specimen.

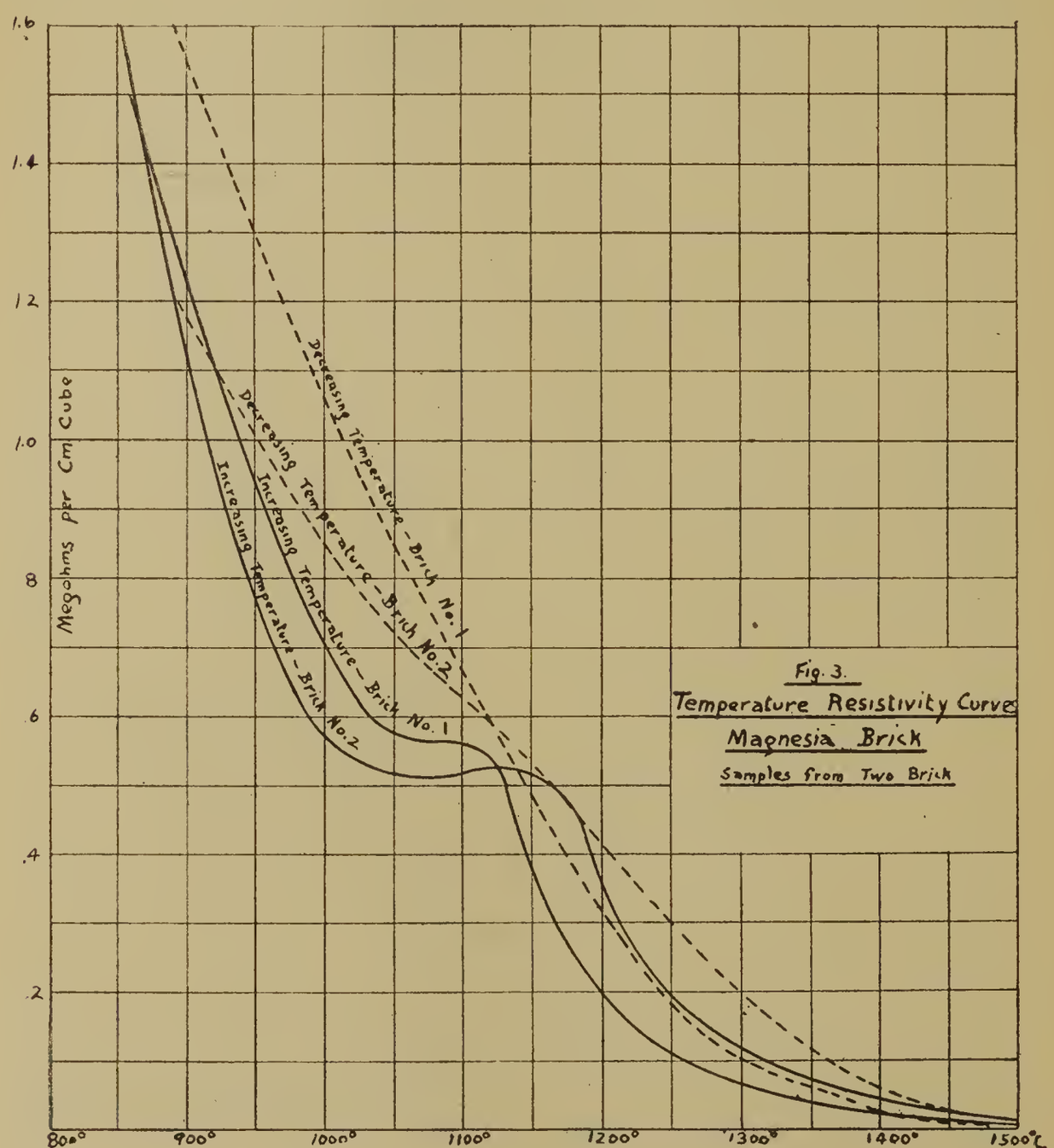


FIG. 3.

2. Bonded carborundum bricks (carbofrax C) containing about 95 percent carborundum have a resistivity comparable to silica bricks, as shown by Fig. 2. The constancy of resistivity with time, when held at 1500° C., is also shown in the upper portion of the diagram.

3. The temperature resistivity curves for magnesia brick are shown in Fig. 3. These bricks were of a dense fine-grained type of standard make. Both specimens showed a peculiar flattening between 1000° and 1150° C. Frequent readings of temperature and resistance were taken in the case of the second brick in order to determine as accurately as possible the exact shape of the

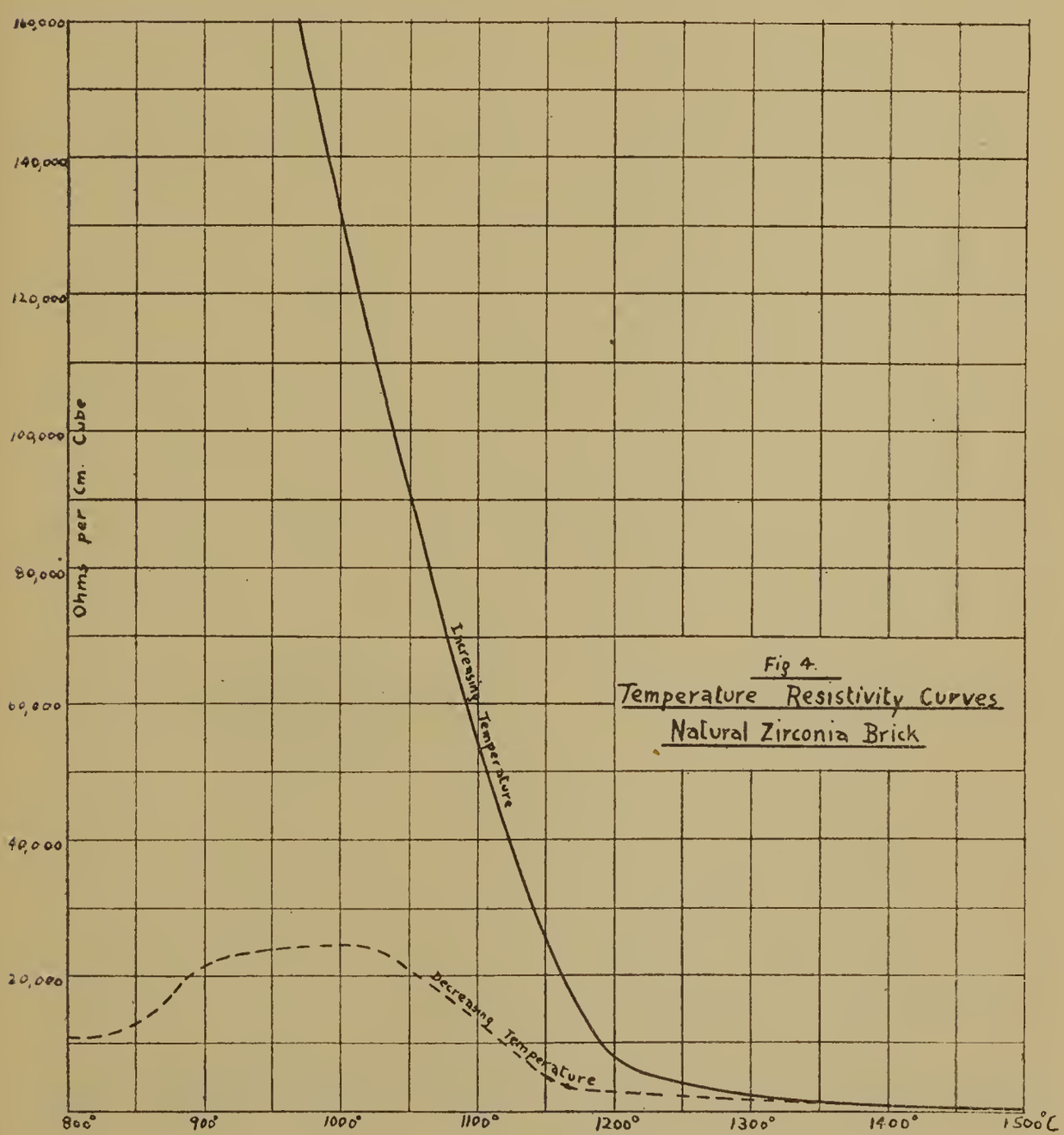


FIG. 4.

curve. The constancy of resistivity between 1000° and 1150° C. indicates either a physical change in the structure of the magnesia at this temperature, as for example crystal transformation, or it may indicate some chemical reaction between ingredients of the brick. The cooling curves did not show this effect in either case.

4. Zirconia bricks made of commercial natural zirconia and containing approximately 80 percent zirconium oxide, changed very rapidly in resistance up to 1200° C. and then decreased

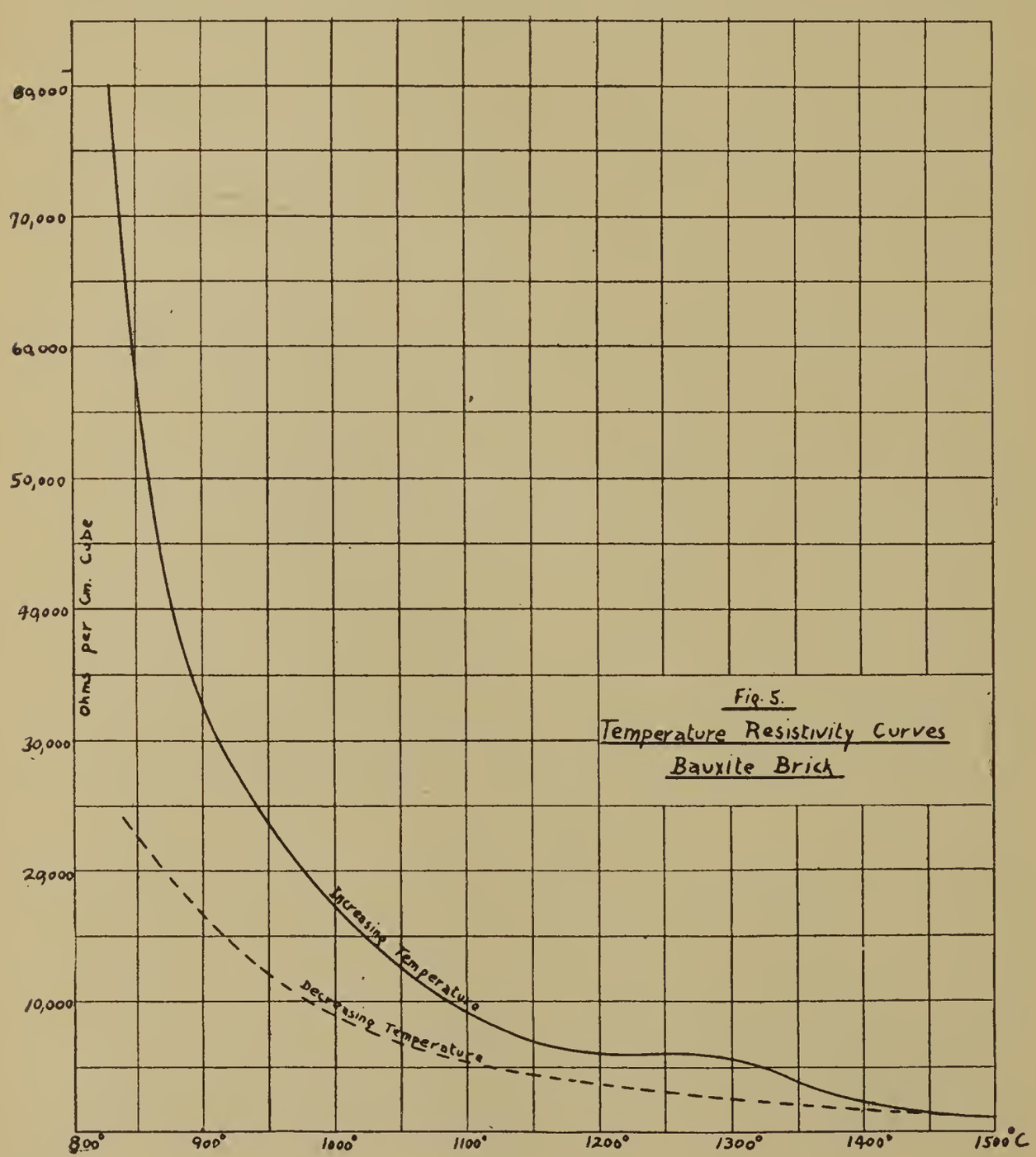


FIG. 5

slowly to 1500° C. as shown by Fig. 4. The irregular shape of the decreasing temperature curve indicates that a permanent change had taken place in the brick, probably in the nature of vitrification.

5. Bauxite brick of a well-known make, coarse in texture (apparent density 1.91), decreased regularly to a low value at 1500° C. Fig. 5. The displacement of the curve for decreasing

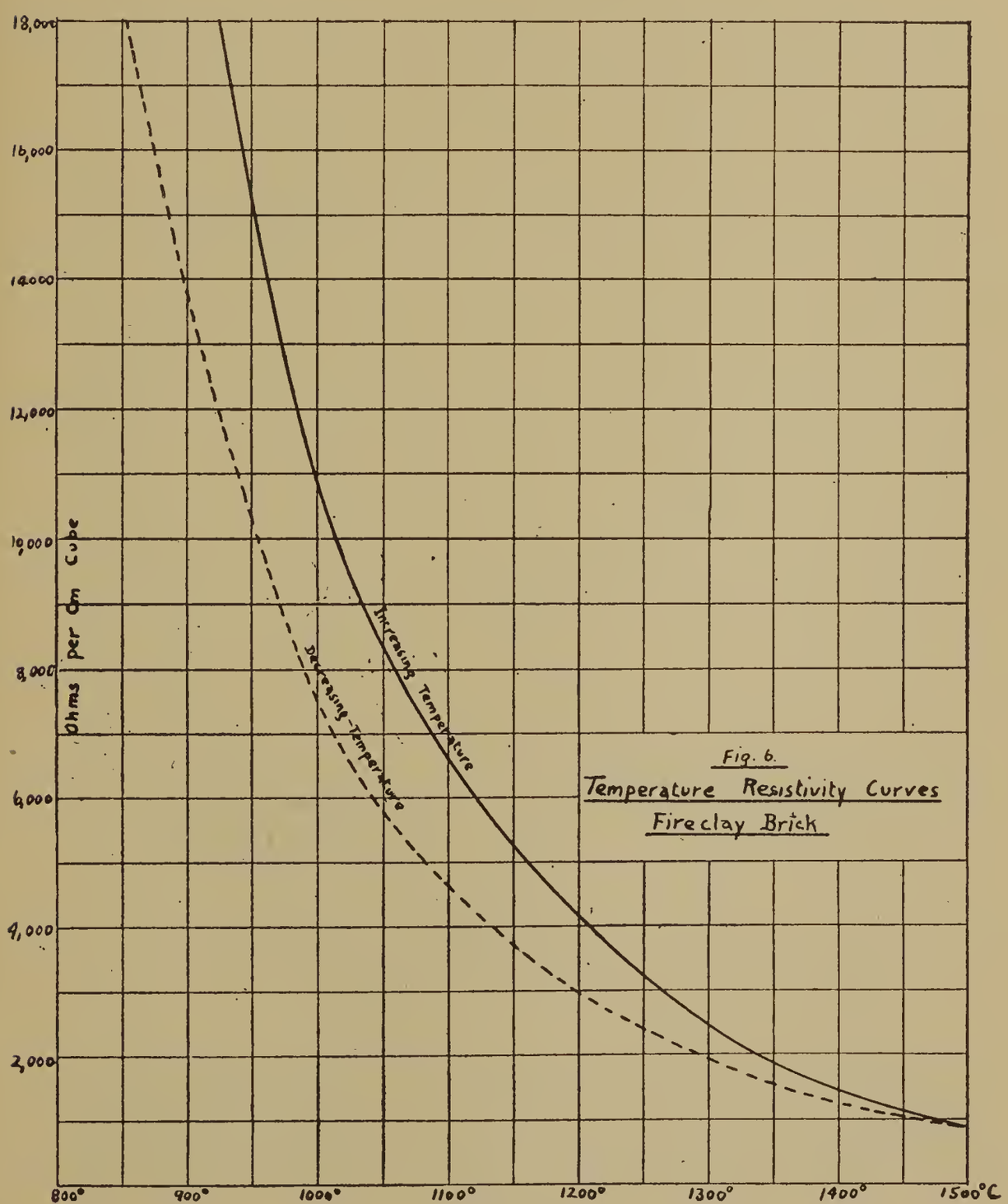


FIG. 6.

temperatures indicates the lag of the thermocouple behind the average temperature of the specimen.

6. Grade A fireclay bricks (apparent density 1.78) very coarse in texture, and highly refractory, gave resistivities as shown by Fig. 6.

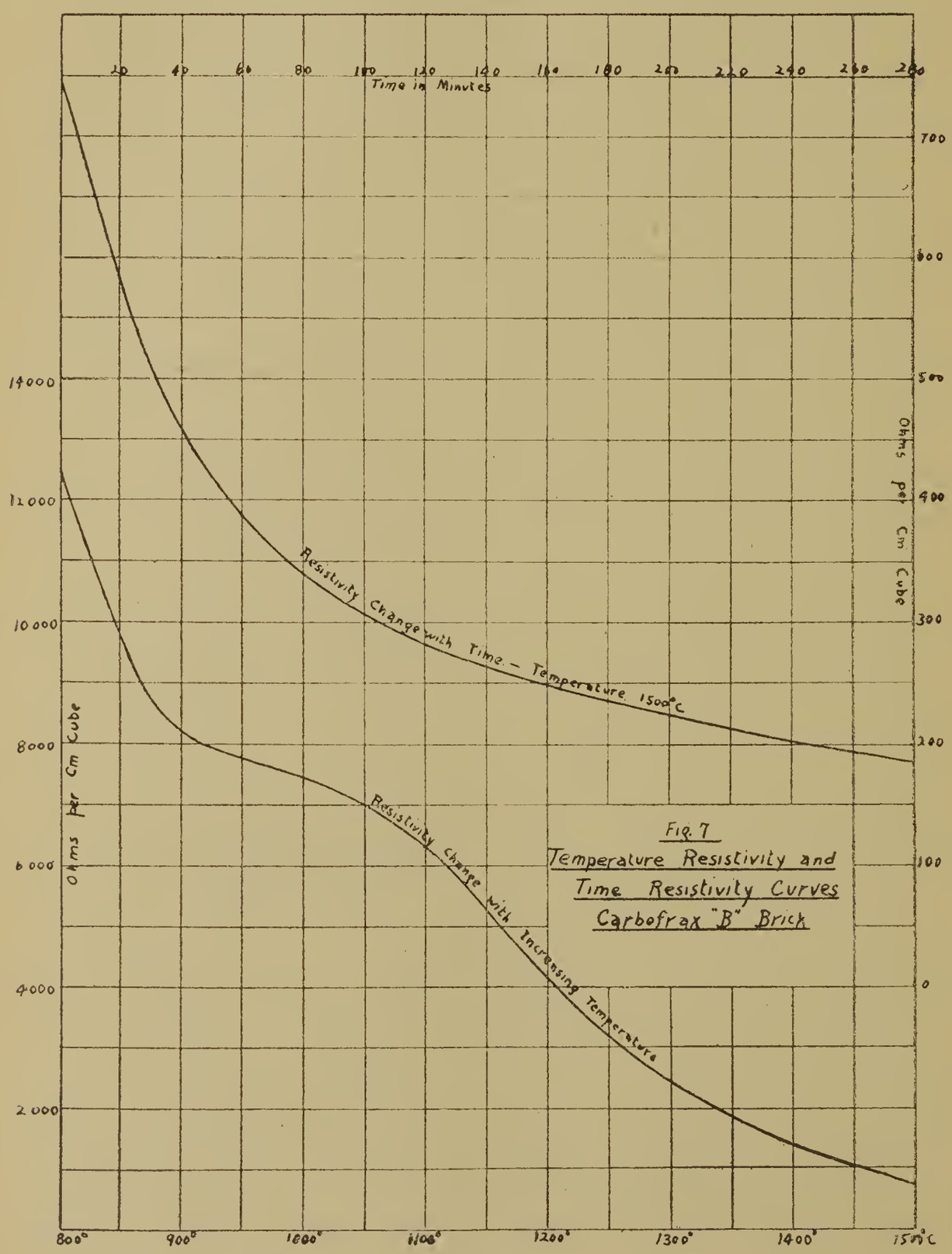


FIG 7.

7. Bonded carborundum bricks (Carbrofrax B), containing about 90 percent silicon carbide, have lower resistance than fire-clay at low temperatures, and approximately equal resistivities above 1000° C. As shown in Fig. 7, there is a rapid decrease in resistivity with increasing time, at 1500° C.

8. Chrome bricks (apparent density 2.09), coarse grained, possess low resistivities at all temperatures as shown by Fig. 8. The specimens all showed a peculiar behavior between 1100° C.

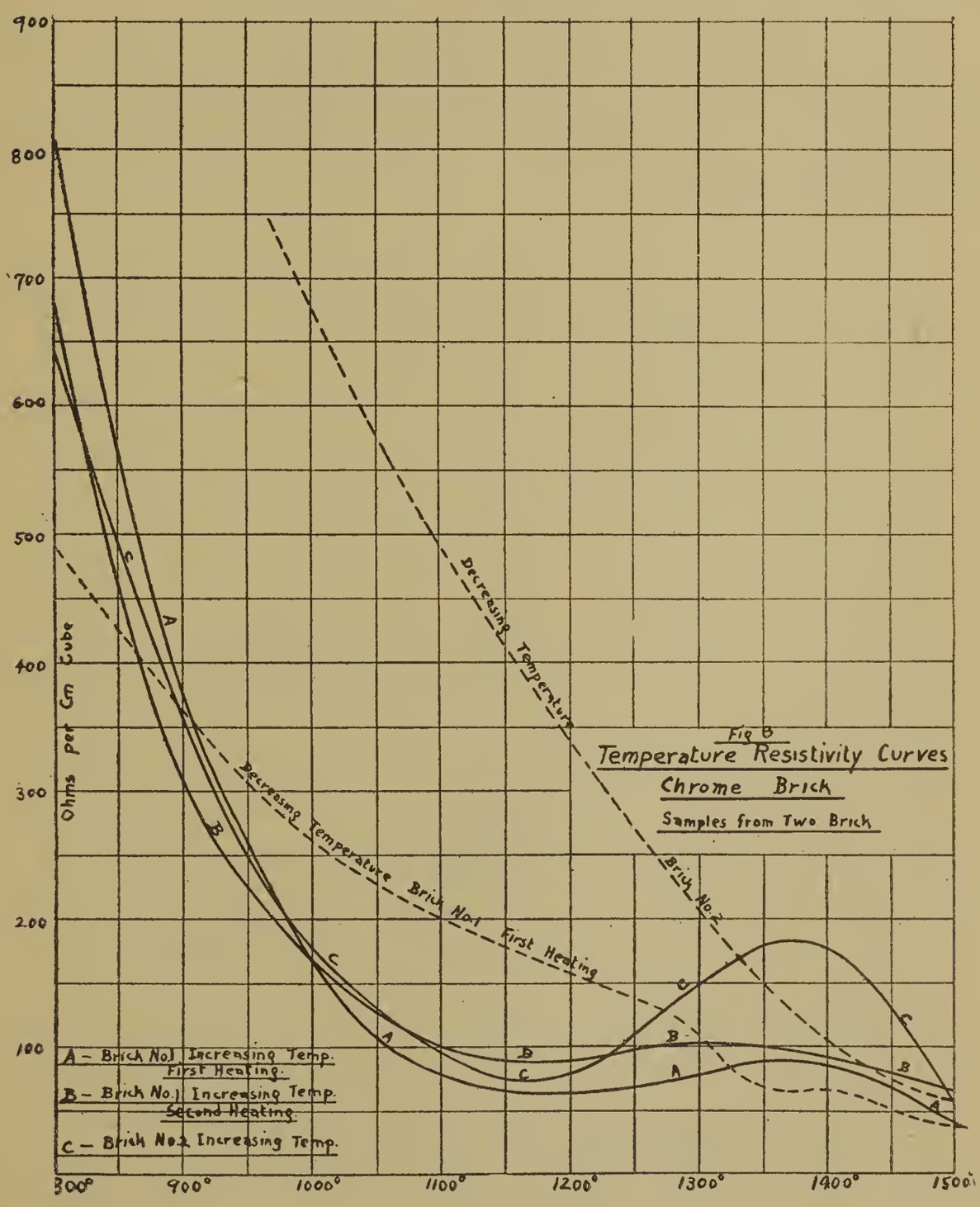


FIG. 8.

and 1500° C. From 1100° to 1200° the resistivity remained practically constant; from 1200° to about 1350° it increased; and from 1350° to 1500° it again decreased. A further peculiarity of this material is shown by the cooling curves which are widely different from the heating curves.

9. Recrystallized carborundum brick ("Refrax") containing no bonding material, has low resistivity at all temperatures. Fig. 9. The change in resistance with temperature is quite regular throughout the range investigated.

The resistivities per centimeter cube at intervals of 100° from 800° to 1500° C. have been deduced from the ascending temperature curves and the results for nine varieties of refractories are tabulated on page 292.

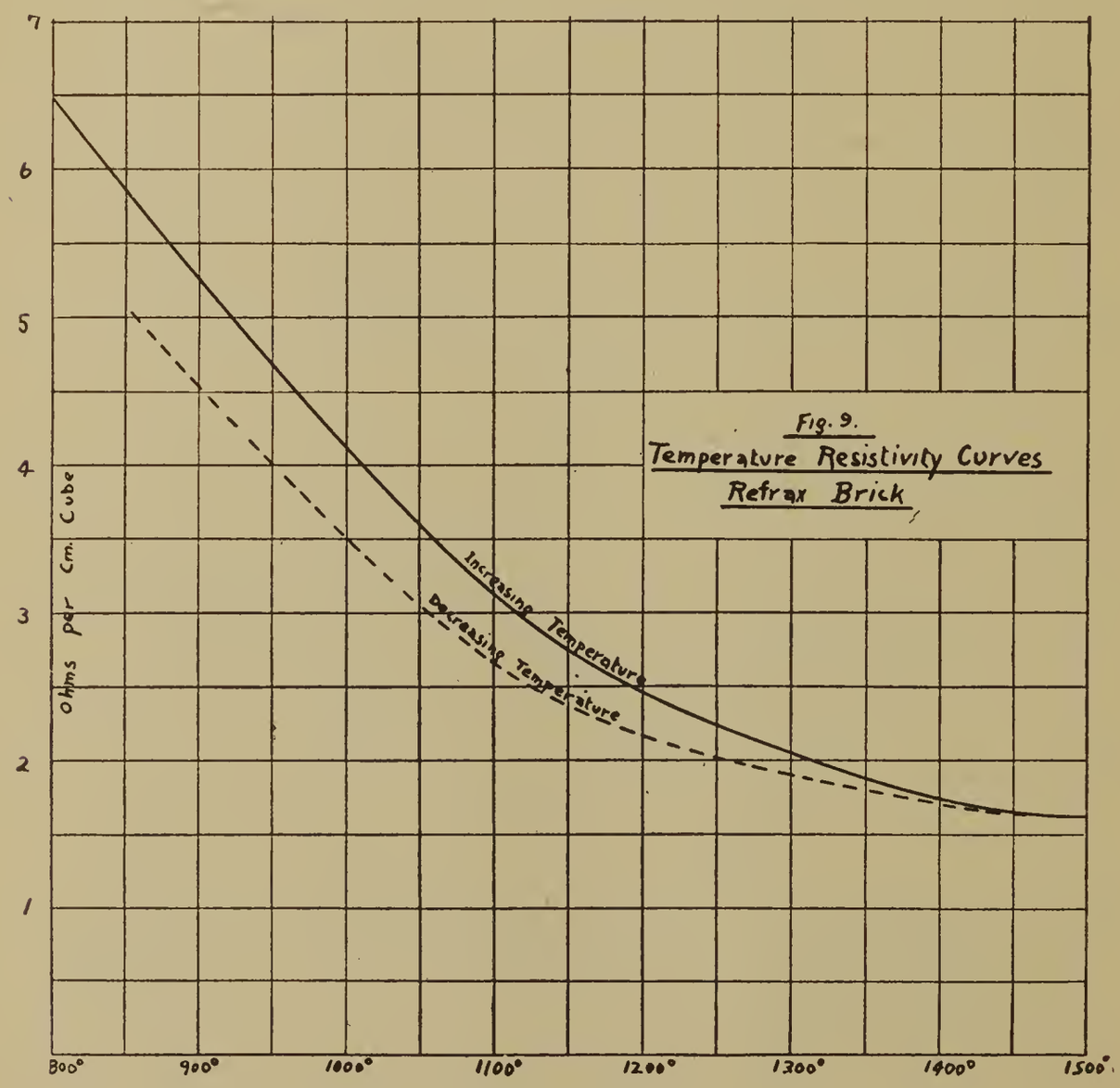


FIG. 9.

These results are plotted on a single scale in Fig. 10, in order to show their relative values.

On account of the variation in the product of one refractory manufacturer to another, or even between two specimens from the same manufacturer, these results cannot be considered as average

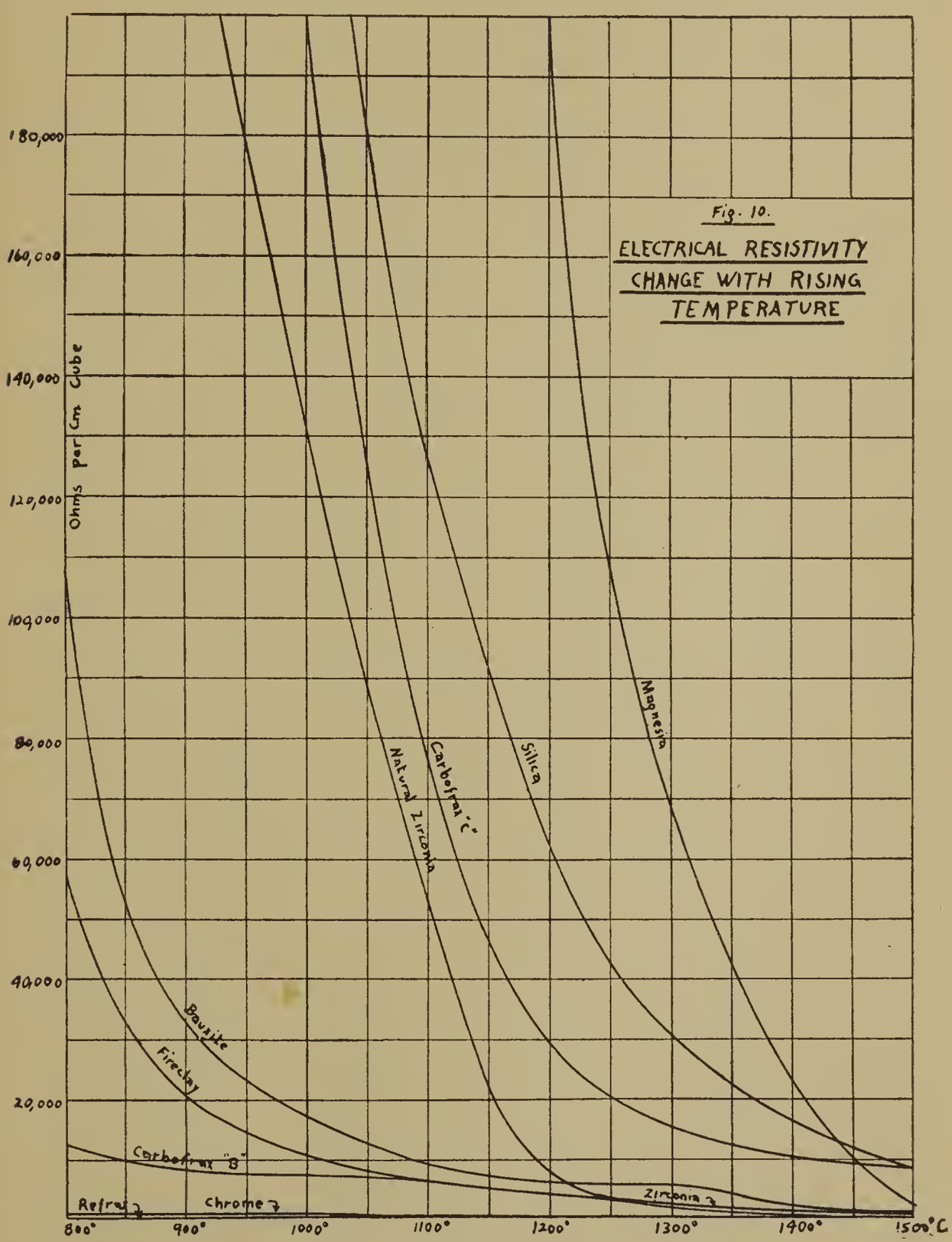


FIG. 10.

*Resistivities of Refractories.*

| | Cold | 800° C. | 900° C. | 1000° C. | 1100° C. | 1200° C. | 1300° C. | 1400° C. | 1500° C. |
|---|---|---|---|---|---|---|---|---|---|
| 1. Silica ..... | < 125 Meg. | 2.38 Meg. | 765,000 | 300,000 | 126,000 | 62,000 | 30,900 | 16,500 | 8,420 |
| 2. Bonded Carborundum (Carbofrax C)..... | < 127 Meg. | 835,000 | 477,000 | 197,000 | 75,000 | 29,500 | 15,200 | 10,100 | 8,590 |
| 3. Magnesia ..... | < 137 Meg. | 5.00 Meg. | 1.24 Meg. | 708,000 | 560,000 | 193,000 | 67,400 | 22,400 | 2,500 |
| 4. Zirconia (Natural) ..... | < 134 Meg. | 558,000 | 224,000 | 131,300 | 53,800 | 7,710 | 2,100 | 968 | 412 |
| 5. Bauxite ..... | < 133 Meg. | 109,000 | 32,500 | 17,200 | 9,200 | 6,100 | 5,600 | 2,200 | 1,100 |
| 6. Fireclay (Grade A).... | < 137 Meg. | 57,600 | 20,600 | 10,800 | 6,590 | 4,160 | 2,460 | 1,420 | 890 |
| 7. Bonded Carborundum (Carbofrax B)..... | 107,200 | 12,550 | 8,220 | 7,420 | 6,320 | 4,160 | 2,420 | 1,435 | 745 |
| 8. Chrome ..... | 48.1 Meg. | 803 ohms | 5.25 | 171 | 78 | 63 | 77 | 85 | 41 |
| 9. Recrystallized Carborundum (Refrax) ..... | 106.9 ohms | 6.45 | 375 | 4.11 | 3.11 | 2.45 | 2.05 | 1.74 | 1.62 |

Note: Data are in terms of ohms per $cm^3$, except where megohms are noted.

values. They do, however, serve to indicate the relative order of magnitude of the electrical resistivities of the different varieties of specialized refractories up to a temperature of 1500° C.

*Research Laboratory,*
*The Carborundum Co.,*
*Niagara Falls, N. Y.,*
*July 21, 1920.*

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## DISCUSSION.

CARL HERING[1]: Such data as these are very useful and valuable to furnace men, if reliable. It seems to me that instead of taking the average of the two curves, the final and useful figures would better have been used on the dotted curves. The dotted curves seem to me to be more important than the others, because there evidently were changes in the material during the first heating. Therefore the full line curve would apply only to a furnace the first time it started, while the dotted line would apply to furnaces during the running periods. In all such measurements, the surface contact resistance may be of considerable importance and possibly may be very high. On the other hand, contact resistance also occurs in practice as a useful thermal resistance. Two bricks laid on top of each other on their flat sides will no doubt have a higher total resistance than if they are laid on edge without a contact resistance between them. Then, in all such materials there is a grave question whether there is not some electrolysis, because such compounds are conductors of the second degree; a counter E. M. F. would then, in these measurements, appear in terms of ohms, and it is sometimes rather dangerous to thus represent a counter E. M. F. The author refers to zirconia; I have been trying to find out what is really meant by zirconia; I find that that term is used by different persons in different ways; it may mean the oxide, the sulphate, or the silicate of zirconium, or a mixture of some of them.

COLIN G. FINK[2]: I should like to ask Mr. Hartmann whether

[1] Consulting Electrical Engineer, Philadelphia, Pa.
[2] Head of Laboratories, Chile Exploration Co., New York City.

he has made any chemical analysis of the bricks he studied. Take a case like magnesia brick; that from one manufacturer will give you entirely different results as compared with that from another. The electrical conductivity is very largely dependent upon impurities. It certainly would add much to the value of the present paper if the authors would include analyses. What applies to magnesia brick applies similarly to zirconia brick; Mr. H. Edwin Coley, of London, E. C., claims that his is superior to the American. It would be interesting to ascertain how much silica was in the bricks Mr. Hartmann tested. This is a very interesting paper; it is an opening wedge into a very important subject; I hope that other papers will follow.

F. A. J. FitzGerald[3]: Has Mr. Hartmann made any determinations of the time effect except on carbofrax materials? Time resistivity curves would be interesting. Have any experiments been made in heating up the second time a magnesia brick, which showed irregularity in heating up the first time, and seeing if the same things occur? That might show whether there is any chemical change. Also, does Mr. Hartmann know at what temperature any of the bricks he worked with were baked? That would cause a considerable variation in the results he gets.

E. R. Morton[4]: In connection with the irregularities in the measurement of carbofrax brick, there are two phenomena with carborundum that are not generally known; one is that it exhibits a striking lateral conductivity. I have had cases that showed a thousand times more in one direction than in the other. Then the heating due to the current which would give somewhat lower resistance. I notice that the measurements are made at 150 volts, which is more than enough to heat the carborundum electrically to redness, from the cold, at the point of contact.

W. S. Landis[5]: This is an extremely valuable paper which opens up a field of research and endeavor that is untouched, I think, by anybody outside of this Society or its members, and I would like to see a very full and free discussion. I think it is going to start a program of work on this subject in a number of places. The question of silica brick is rather an interesting

[3] FitzGerald Laboratories, Inc., Niagara Falls, N. Y.
[4] New York City.
[5] American Cyanamid Co., New York City.

one, because they have been so widely used in the construction of certain parts of electric furnaces. The silica bricks, as you buy them, and after they have been heated up for some little time, are two decidedly different things. The Geophysical Laboratory at Washington has done considerable work on the transformation of silica to tridymite. I would like to see experiments made on such brick after it had been heated up a week or two in the electric furnace. The deflection in that conductivity curve of the magnesia refractory is largely due to large quantities of iron oxide in magnesia bricks. If that had been repeated on a magnesia brick, according to my own experience it would not have occurred during the second heating.

L. E. SAUNDERS[6]: I agree with the others, that this is a very important contribution to our practical knowledge of refractories, and the thing that strikes me first in connection with it is that it is too bad that more than one specimen of a kind was not taken. It is so important that we have this knowledge, that I hope the authors will repeat their work, getting their specimens from different sources, and at the same time giving us a little more information on what the specimens consist of. In connection with the magnesia brick, it is very important to know the origin and nature of the impurities. The old Austrian magnesite bricks were made of magnesia found in nature containing iron oxide. The more modern magnesia bricks are made from relatively pure magnesium oxide to which varying quantities of iron oxide have been added, and I think all will agree that the resistivities would be different in the two cases, and there would be very possibly a reason for these humps in this magnesia curve.

A. M. HUNT[7]: I have read this paper with a great deal of interest, and its discussion has brought up in my mind a thought that has occurred to me many times before, viz., that if the Society would appoint a committee or committees to outline the type of data that would be desirable in connection with much of the experimental work which is done, the results would be of much more value to the profession at large.

J. W. RICHARDS[8]: This paper illustrates very well the distinc-

[6] Norton Co., Worcester, Mass.
[7] Consulting Engineer, New York City.
[8] Prof. of Metallurgy, Lehigh University, Bethlehem, Pa.

tion between pure scientific investigation and practical engineering investigation. If we want to find out the actual properties of pure materials and deduce from them the laws of Nature, we must work with the purest materials obtainable. The results are the basis of an investigation of natural laws. If we work with the materials which are available in engineering, they are usually so impure that we cannot deduce from them general laws, but get results which are true only for the material tested; *i. e.*, one of the materials which the engineer has to work with. In this paper, it is to be regretted that no analyses of the materials are given.

The paper is otherwise a valuable contribution, but it is our duty here to point out ways in which it can be made still more valuable.

The method used by the authors in reaching their results is all right as far as their experiments are concerned, but it is highly desirable that the conductivity and resistivity of these materials be determined after they have reached a constant value at the temperature named. The table on page 292 gives the resistivities of the materials when they have been heated at a certain rate to a certain temperature; but some of these materials afterwards decrease in resistivity 25 percent to 75 percent, if they are held at that temperature. For practical purposes, what we want to know is the final resistivity which they acquire at a given temperature indefinitely prolonged. As they stand, the results are interesting but not applicable to actual electric furnace problems. The proper way to extend this research and make it applicable to the actual electric furnace problem, is to take samples and keep them a day or a week at each given temperature until their resistivity has stopped changing, and then catalogue the result as their real resistivity at that temperature. Let me suggest to the authors that they may find around some works bricks which have been heated to a certain temperature for a long while, such as an open hearth furnace where the bricks have been kept at an average temperature of 900 or 1000° C. perhaps for a week, and by getting samples of these they may get their final results at once. They could find electric furnaces that had been running for months.

One can thus get results which would be applicable to the furnace in its running condition.

I have been trying to surmise why the electrical resistivity of these materials decreases so tremendously. That is something on which we may profitably speculate. I cannot help but think that one cause may be that the pores of the material become filled with metallic vapors. Take carborundum, for instance; we know that it dissociates into graphite and silicon at perhaps 2,300° C. under atmospheric pressure, and undoubtedly it has some dissociation tension at 1,500° C.; this will fill the pores of the material with silicon vapor. We may then have the conductivity of silicon vapor to help carry current. We know that the mercury arc is a current through mercury vapor and that the zinc arc conducts very well. It may be that there are some metallic vapors filling the pores of these materials and reducing the resistivity at high temperatures.

Carl Hering: I think Dr. Richards' last surmise is probably not correct, because the same thing is true of glass, and there are no pores in glass; we know that the resistivity of glass falls enormously with an increase of temperature.

C. P. Madsen[9]: Several years ago I made a large number of approximate measurements on refractory materials from which several hundred curves were plotted. The work was intended to determine the value of refractory dielectrics for domestic heating apparatus, and therefore resistivity only up to 1,000° C. was measured. The results obtained, however, may answer some of the questions asked. The resistivity was measured on both rising and falling temperature. The general conclusions are:

(1) The resistivity of silicates decreases many times faster than that of oxides of the same metal.

(2) The presence of small amounts of impurities increases the resistivity decrease very much on temperature rise.

W. G. Harvey[10]: I could not help but agree with Dr. Hering on the importance of the descending or dotted curve. I would like to ask Mr. Hartmann if, in doing the work, he had occasion to repeat the work on each sample twice, and whether the curve checked or did not check?

[9] Consulting Engineer, New York City.
[10] Supt. and Metallurgist, American Magnesium Corp., Niagara Falls, N. Y.

M. L. HARTMANN: In some cases the measurements on carborundum bricks were repeated on the sample twice and the curve practically checked, and finally it was held at one temperature.

CARL HERING: Which one of the two curves?

M. L. HARTMANN: The dotted curve.

CARL HERING: Did you repeat it on the magnesia brick?

M. L. HARTMANN: The measurements on the magnesia bricks were not repeated.

C. P. MADSEN: Some further conclusions from the work I have already mentioned may throw further light on the questions asked. My work was done on smaller samples and the apparatus was such that the thermal lag was exceedingly small. I was therefore able to check the difference between the resistivities on rising temperature and those on falling temperature. In general pure oxides checked. Some silicates did; but mixed oxides and silicates never checked, neither did materials containing appreciable impurities.

M. L. HARTMANN: The paper as given is admittedly simply exploratory, and I think the search that has been outlined by the criticism of the paper could be well undertaken by the Bureau of Standards or some large institution of that kind rather than by an industrial laboratory. We realize that many things are imperfect in the paper. As to the criticism in connection with the analyses of the specimens—we will endeavor to add those analyses to the written discussion of the paper. The question of doing more samples, that Mr. Saunders brought up, and getting an average, was considered, but we felt that inasmuch as this was simply a preliminary paper, and, as I said, an exploratory paper, it was hardly worth while for our specific purposes to gather in a large number of samples. The question of holding bricks at 1,500° C. for two weeks in an electric furnace, as was suggested in the discussion, would be rather difficult with the type of furnace we used, and the only way of getting bricks which had been heated for a long while at that temperature would be as suggested by Dr. Richards, to collect them from various commercial furnaces.

L. E. SAUNDERS: I hope Mr. Hartmann will not give us

average results from a series of specimens, but the whole series of results.

M. L. HARTMANN (*Communicated*): As stated before, this investigation was purely an exploration into a field about which there was little or no published information. We wished to know how the resistivities of carborundum refractories compared with those of other specialized refractories, this being incidental to a larger problem in our Research Laboratory.

The great need for data concerning the electrical resistivities of commercial refractories is evidenced by the discussions, which have appeared mainly to point out the information which each member would like to have done for him, in a comprehensive investigation of the subject.

Dr. Hering's criticism concerning the data in the table is based upon an incorrect premise, inasmuch as the data are taken only from the ascending temperature curves, and are not average values from the two curves. In practical electric furnace work we seriously doubt whether the descending curves would be of any more value than the ascending curves, because in those bricks whose resistivities on descending temperatures are markedly different from the ascending temperature values (namely, zirconia, bauxite and chrome) there have been undoubtedly very great changes in composition within the bricks. These transformations would hardly be expected to be completed in one short heating—hence these irregular curves merely indicate a system out of equilibrium, and the actual values are not significant. If it were feasible to keep these specimens at the maximum temperatures until equilibrium had been obtained, then the descending temperature or dotted curves would indicate the true resistivity. On the other hand, those materials which increased in resistance on cooling at nearly the same rate as they decreased on heating, are evidently somewhat stable systems and large changes of resistance with time would not be expected, so long as the material was not contaminated by slags or vapors.

In practical work it would be extremely difficult to secure brick which had been kept at high temperatures for a long time, and which had not during this time become penetrated by vapors and slags, and hence the resistivities of these would have

little value in furnace design. For the same reasons we do not believe that the suggestion made by Dr. Richards is practicable. To secure specimens of this wide variety of refractories, which had been subjected to comparable heat, slag and vapor treatment would be difficult; and the resistivities would be useless unless these conditions were comparable. If one wished to know the resistivities of all of these refractories after they had been subjected to a given set of conditions—for example, in the roof of an electric steel furnace—such data would be valuable in designing furnaces for those conditions but would be useless for other furnace conditions. Such studies were entirely outside the scope of the present paper.

We do not believe that the phenomena mentioned by Mr. Morton affected the measurement of the resistivities of the carborundum bricks. The crystals of silicon carbide are distributed in every possible orientation in a brick, and the measured resistance would represent the composite or average resistance. The measuring current at 150 volts was not allowed to flow long enough to heat the crystals, even at points of contact. Furthermore, in the bonded carborundum refractories, the carborundum particles are held together by a ceramic material which partly prevents electrical contact.

In reply to Dr. Fink's inquiry, the chemical analyses of the specimens measured are given below:

| | $SiO_2$ Percent | $Fe_2O_3$ Percent | $Al_2O_3$ Percent | CaO Percent | MgO Percent | $TiO_2$ Percent | Miscellaneous Percent |
|---|---|---|---|---|---|---|---|
| Silica..... | 94.8 | 0.2 | 1.8 | 2.4 | 0.3 | ... | |
| Magnesia..... | 6.0 | 0.3 | 6.3 | 3.1 | 84.1 | ... | |
| Zirconia, Natural | 21.5 | 0.7 | 4.7 | 0.0 | 0.0 | 0.5 | $ZrO_2$=72.6 |
| Bauxite..... | 40.4 | 0.9 | 55.5 | 0.2 | 0.0 | 2.7 | |
| Fireclay..... | 50.4 | 0.8 | 45.5 | 0.5 | 0.8 | 1.9 | |
| Chromite..... | 15.8 | 13.2 FeO | 7.3 | ... | 18.5 | ... | $Cr_2O_3$=40.9 $Mn_3O_4$=18.5 |

*A paper presented at the Thirty-eighth General Meeting of the American Electrochemical Society, in Cleveland, September 30, 1920, President Landis in the Chair.*

# DETERIORATION OF NICKEL RESISTORS.

By F. A. J. FitzGerald[2] and Grant C. Moyer.[2]

## Abstract.

A record of attempts to use pure nickel wire as a furnace resistor in place of nichrome wire. It was found to have become brittle and to break easily. This was investigated, and the cause found to be in the absorption of sulphur. When sulphur is absent from the materials with which it is in contact, and from the atmosphere of the furnace, pure nickel wire can be substituted for nichrome resistors in some cases. [J. W. R.]

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There are two objections to the use of "nichrome" wire as a resistor in electric furnaces. First, the relatively minor one, its high cost; second, the onerous conditions attached to a license for its use. It seemed possible that for some purposes nickel wire might prove to be a suitable substitute in spite of its high thermal coefficient of electric resistance, for this disadvantage can be overcome, and its deterioration by oxidation, since this, with reasonable precautions, can be diminished so as to give a fairly extended life.

It was found, however, in some heating devices that nickel resistors broke down after very short service, for although there was no appreciable oxidation the resistor wire had become so very brittle that the slightest bend would break it. The resistor wire, in the particular furnace we had made, was wound on a form embedded in a refractory cement and heat insulated with infusorial earth. The cement and heat insulator both contain carbonaceous matter and it was thought probable that a carbonization of the nickel was the cause of the brittleness. Some of the nickel wire which had become brittle was placed in a boat in a combustion furnace and heated in a current of oxygen, which,

[1] Manuscript received August 2, 1920.
[2] FitzGerald Laboratories, Inc., Niagara Falls, N. Y.

after leaving the combustion tube, was passed through a solution of barium hydrate. This gave a distinct precipitate of barium carbonate, which seemed to be confirmation of the conclusion we had reached. However, we were wrong, and it may be worth while to describe how we found this out.

In order to investigate further the effect of heating nickel in the presence of carbon monoxide, some pieces of soft nickel wire were put at one end of a porcelain boat while in the other end were some pieces of carbon rod 0.3 mm. in diameter. The boat was then put in the tube of a combustion furnace, the ends of the tube tightly closed and the tube heated for 24 hours at 770° C.; the wire was found to be brittle.

It appeared, therefore, that if soft nickel wire was heated in the presence of carbon monoxide for 24 hours at a temperature of 770°–780° C. it would become brittle, and this apparently explained why the resistor in our furnace failed, since the wire certainly reached therein a temperature of 800° C.

Further investigations were then undertaken to determine the conditions under which nickel wire becomes brittle in presence of carbon dioxide and these tests showed that our conclusions were wrong.

Nickel wire embedded and heated in powdered willow charcoal for 19 hours at 600° C.: Wire soft.

Heated for 23 hours at 730° C.: Wire soft.

Heated for 39 hours at 800°–830° C.: Wire soft.

Heated same nickel wire for 48 hours at 740°–760° C. with some pieces of the carbon rod used in previous experiments: Wire very brittle.

It was evident from these experiments that the cause of the deterioration of the resistor wire was not due to heating in presence of carbon monoxide; but to something else. It seemed probable that sulphur was the cause of the trouble because it was known that the carbon rod contained a small percentage of sulphur, while the willow charcoal probably did not.

Two porcelain boats were put in the combustion furnace, tightly closed at both ends. In one boat were pieces of commercial iron sulphide (FeS), in the other some pieces of nickel wire. Heated at 820° for 19½ hours: Wire very brittle.

Similar test, but temperature 620° for 18 hours: Wire very brittle.

Similar test, but temperature 530° for 46½ hours: Wire very brittle.

Similar test, temperature 520° for 18½ hours: Wire somewhat more brittle than original. Three samples, bent first at right angles then bent back through 180°, broke in 3, 4 and 2 bends respectively, while three samples of original wire broke in 7, 6 and 6 bends respectively.

A number of pieces of soft nickel wire were put in one end of a porcelain boat with a piece of iron sulphide at the other end. The boat was then heated in the closed combustion furnace at a temperature between 500° and 520° C., with the following results:

At end of 24 hours piece of wire removed and found to be brittle, broke in 2 bends. The fracture showed that the outer layer of the wire was changed in appearance, due presumably to penetration of the sulphur.

At the end of 48 hours, specimen of wire broke with 1 bend. Fracture showed further penetration of sulphur as indicated by changed appearance of metal, the central core of apparently unchanged nickel being now considerably less than half the diameter of the wire.

At the end of 96 hours, the wire was so brittle that the least bend broke it and the central core was very small.

At the end of 120 hours the core was just visible.

Some experiments were made to determine if the wire could be made soft again by heating in a current of air. Accordingly samples of brittle wire were heated in the combustion furnace at 600° C. in a current of air for 23 hours. The wire was still brittle; but when heated again under the same conditions for 47 hours at 700° C. the wire became soft, breaking in 5 bends.

In order to find out whether or not there was any action of sulphur on nickel at low temperatures, pieces of nickel wire and small pieces of sulphur were sealed in glass tubes that were drawn out so as to contract the area of the bore at one point, the sulphur being thus confined at one end of a closed tube, and the nickel wire at the other. Some specimens of nickel were also embedded in powdered sulphur in a test tube. The sealed tube was put in

an oven heated to 90° C. to 100° C., the test tube was put away in a drawer in the laboratory.

At the end of a week the wire in one of the closed tubes was examined and found to break in 3 bends. At the end of 3 weeks another specimen was examined, but did not appear to be more brittle than the first one examined.

The wire packed in the powdered sulphur when examined after 10 days broke in 3 bends, at end of 20 days broke in 3 bends, at end of 41 days the same.

Finally it may be noted that when nichrome is heated at high temperatures in the presence of sulphur it also tends to become brittle; but the effect is by no means as marked as in the case of nickel wire. This was shown by the following experiment:

Some pieces of nickel wire breaking on the average in 6 bends and some nichrome wire breaking on the average in 4 bends were heated in presence of iron sulphide at 740° C. for 18 hours, after which it was found that the nickel wire broke in 1 bend and the nichrome in 2 bends.

Finally some experiments have been made with a small crucible furnace using nickel wire as a resistor and taking care to have cement and heat insulation free from sulphur. The nickel resistor stood up well in these furnaces.

## CONCLUSIONS.

Nickel wire will not make a good resistor in electric furnaces where it is subject to the action of sulphur.

Nickel wire heated in the presence of sulphur becomes brittle.

Sulphur makes nickel wire brittle even at low temperatures, and the effect is very marked and rapid at temperatures above 500° C.

The action of sulphur in causing brittleness is also found in the case of nichrome wire, but the effect is not as marked as in the case of nickel wire.

Nickel wire made brittle by sulphur may be softened by heating in an oxidizing atmosphere.

Nickel wire seems to stand up well as a resistor when precautions are taken to prevent reaction with sulphur.

*FitzGerald Laboratories, Inc.,*
*Niagara Falls, N. Y.*

## DISCUSSION

F. A. J. FitzGerald: The original nickel wire used was perfectly free from sulphur. The other nickel wires that showed brittleness both contained sulphur. I had some analyses made of the nickel, and we found that the nickel wire used in the small electric furnace contained 0.106 percent of sulphur, and the nickel wire heated at 500 to 520° C. for 96 to 120 hours in the presence of iron sulphide contained 0.054 percent sulphur.

C. P. Madsen[1] The onerous patent situation named makes this paper of unusual interest to the research engineer. It is regrettable that the data given are meager, and, while the author has just given analyses of the sulphur content of the material used, the lack of a complete analysis makes the results inconclusive. The physical properties of nickel are influenced very much more by the presence of small impurities than any other metal and commercial analysis cannot be depended upon. My opinion is that the specimens used were poor samples, although definite conclusions cannot be drawn in the absence of the size of wire used and exact manner of making bends. I made some tests on 99 percent sheet nickel 0.005 inch (0.125 mm.) thick and found that it would stand from 14 to 16 relatively sharp bends, while pure nickel of the same thickness, made in my own laboratory, stood about 50 bends.

It is well known that the presence of sulphur in nickel makes it brittle, but it is not so well known that it will absorb enough sulphur at relatively low temperatures to cause this trouble, and this is, therefore, an important contribution to the art.

The author's method of determining the influence of carbon, however, does not permit of drawing conclusions, but, on the other hand, prior literature on the effects of carbon with relation to other impurities upon the physical properties of nickel is meager. It is claimed by some that a very small amount of carbon increases the ductility of the metal, and a further increase makes it brittle, while a still further increase makes it ductile again. These effects, however, are influenced very much by the presence of small amounts of other elements, notably manganese and oxygen.

[1] Consulting Engineer, New York City.

While it is true that under some conditions CO gas does not embrittle nickel, and is, in fact, used commercially for annealing, the fact remains that under some conditions it does embrittle nickel. This again may be governed by relative amounts of O and CO and by temperature points related to nickel carbonyl. I therefore feel that much more work must be done on the effects of carbon upon the physical properties of nickel with relation to other impurities before any definite conclusions can be drawn.

It might be of interest to know, however, that the addition of about 2 percent. chromium has a tendency to offset the injurious effects of other impurities, and the addition of this amount of chromium has been construed to not come under the Marsh patents.

Colin G. Fink[2]: I should like to ask Mr. Madsen as to what influence 2.0 percent manganese has on the removal of the embrittling effect.

It has been our experience that manganese offsets brittleness.

C. P. Madsen: Replying to Dr. Fink's question, I hesitate to make any definite statements about nickel. As you probably know the literature on this subject is very meager, and, although my research has been quite elaborate, I do not yet feel that I have reached any final conclusions. My results to date, however, indicate that manganese is not as effective as chromium in overcoming the deleterious influence of other impurities, especially in the presence of carbon. The reason, probably, is that manganese forms a relatively stable carbide which segregates more or less, diminishing the malleability of the metal at normal temperatures. It is, however, added to commercial nickel which is to be hot rolled, but I do not know whether it is of actual benefit in this case, and if so, why.

F. A. J. FitzGerald: The wire that we used in the experiments was No. 16 (1.3 mm. thick). I did not say that sulphur was the only cause of brittleness; I said it was one of the causes. We found out, I think, that carbon undoubtedly does have an effect; in fact, I consulted a metallurgist who had had a good deal of experience with nickel, and it was he who originally suggested that it was the effect of carbon monoxide, and it probably does have

[2] Head of Laboratories, Chile Exploration Co., New York City.

some, but it is small compared to sulphur. However, I did not mean to imply that sulphur was the only cause. We found that nickel, if we took good care, could be used, for a time at least, with satisfaction and without having to comply with the requirements involved in the case of licenses for nichrome.

J. W. RICHARDS[3]: There is one point in the paper which has interested me very much, and that is that nickel wire packed in sulphur and left at ordinary temperature absorbs sulphur and becomes brittle. It is a remarkable fact that with the vapor tension of the sulphur so low at those temperatures, the nickel will absorb it. We have other examples of metals absorbing the vapors of other materials at temperatures below the melting point, such as the Sherardizing process, where zinc is absorbed by iron; also packing it in iron sulphide, the dissociation of the latter is apparently sufficient to give off sulphur sufficiently to sulphurize the nickel.

F. A. J. FITZGERALD: We have another good example of the formation of metallic sulphides at low temperatures in the experiment of carrying a rubber band in the same pocket as a silver watch.

[3] Professor of Metallurgy, Lehigh University, Bethlehem, Pa.

*A paper presented at the Thirty-eighth General Meeting of the American Electrochemical Society, in Cleveland, September 30, 1920, President Landis in the Chair.*

# SOME PHENOMENA OBSERVED IN ELECTRIC FURNACE ARCS.[1]

By J. Kelleher.[2]

As electric furnace loads increase, the regulation or maintenance of these loads at a uniform value becomes of great importance. There are at present many different automatic regulators, each having certain advantages. No matter how well any one of these regulators is adjusted, much is still to be desired. To those who have much to do with furnace regulation it soon becomes evident that the movement of the carbons or electrodes is not the only factor tending to cause variations of load in an arc furnace. It will be observed that in large ferro-alloy furnaces, and especially those furnaces where the charge consists of second-class conductors, the movement of the electrodes has to be considerably slower than in a steel furnace, for example, to prevent hunting.

Another peculiarity is often found in three-phase furnace work: Although the current flowing to the furnace is apparently constant, the regulators often act upon the electrodes, unbalancing the load. The Thury regulator, which moves the electrodes through a small distance at regular intervals of time, has, as the load of the furnace approaches the normal, a peculiar hesitating action; that is, the intervals of time between the movements of the electrodes become greater, as if the regulator waited for the furnace to reach the normal load without further assistance. This peculiar action led to the theory of a furnace "time element."

By "time element" is meant that in a certain furnace, if the electrode is moved through unit distance, it will take a certain length of time for the current to become stable. This length of time may vary for different materials, temperatures, etc.

In order to study the time element, a number of experiments were made in the electrochemical laboratories of the University

[1] Manuscript received August 21, 1920.

[2] Demonstrator and Research Assistant, Faculty of Applied Science and Engineering, University of Toronto.

of Toronto and the FitzGerald Laboratories, Niagara Falls, N. Y., to ascertain if this time element really existed and, if so, how it was affected by material, temperature, etc.

During the winter of 1919-1920, at the University of Toronto, preliminary experiments were carried out on a Siemens single-electrode furnace employing direct current, the main object of these experiments being to determine the effect of slags of different compositions upon the current variation caused by a given movement of the electrode. The average power used in the furnace was 20 kw., and the electrodes ranged from 2 inches (5.1 cm.) to 4 inches (10 cm.) in diameter, both graphite and carbon

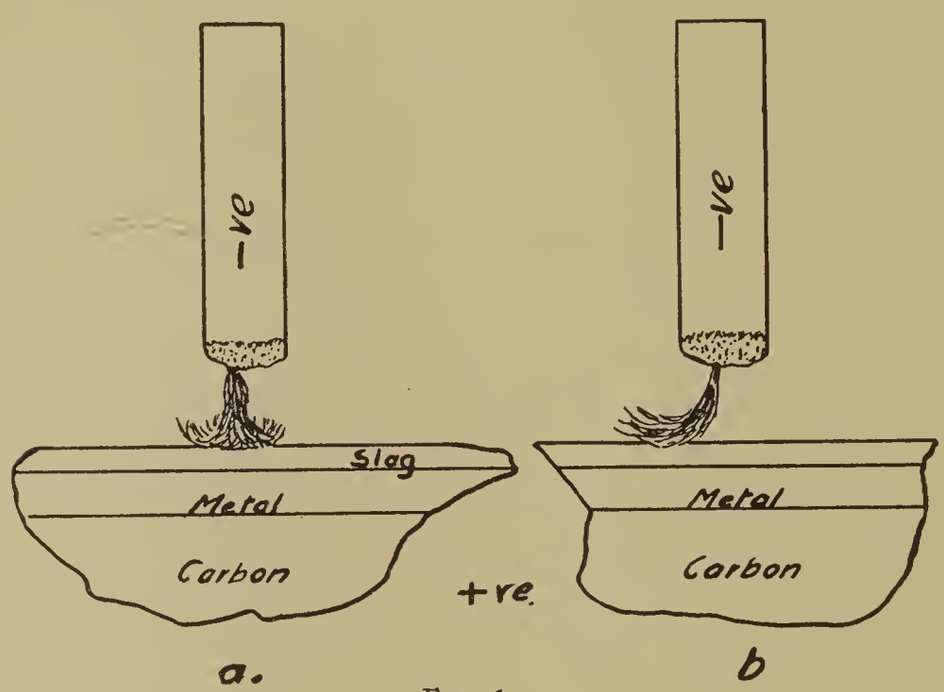


Fig. 1.

being used. Due, however, to the difficulty of maintaining constant potential across the furnace terminals, the results were rather confusing, and perhaps even untrustworthy. It was during these experiments that certain phenomena were observed which appear to be worth recording at this time, although the work planned has not been completed.

A bath of iron-nickel alloy covered by an acid calcium silicate slag was melted in the furnace. To observe the arc under these various conditions the following arrangement was employed: An opening was made in the front wall of the furnace. The opening was then covered by a piece of sheet iron in which a pinhole had been drilled. The light emerging from the opening was allowed to fall on a ground glass screen, which was moved back-

ward and forward until a clear inverted image of the arc was obtained, thus permitting sketches and even photographs of the arc to be made. A few of the sketches made are shown in diagram form. Fig. 1, *a* and *b,* represents what will be called a normal arc, that is, one where the movable electrode is the negative pole. The flame apparently flowed from the electrode to the slag, depressing the slag and flaring out to all sides, as in the case of diagram *a,* Fig. 1, or to one side in the case of *b,* Fig. 1. An arc length of about 3 inches ((7.6 cm.) could easily be maintained. Under these circumstances the arc was silent.

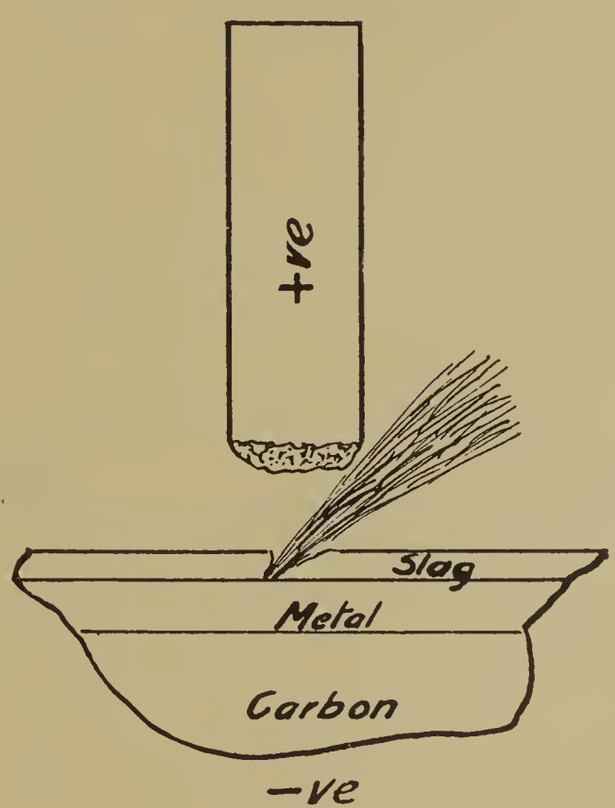

FIG. 2.

Fig. 2 represents what occurs as soon as the polarity of the furnace is changed. A very unstable arc appeared. This arc started below the surface of the slag, the flame moving away from the slag surface and projecting particles of slag into the air with considerable force. The length of this arc could barely be maintained at a greater length than 1 inch (2.54 cm.). The noise of the arc was loud and spluttering.

It had been noticed that if, with a normal arc such as *a* and *b,* Fig. 1, large currents were allowed to flow, the arc became noisy and had a tendency to quench itself, even at comparatively short lengths. What actually happened is seen in Fig. 3. Two distinct

arcs were formed, as shown at *c*, Fig. 3. One of these arcs was normal, as shown in Fig. 1; the second arc was formed as in Fig. 2. As long as these arcs remained separate, a good arc length could be maintained. Heavy brown fumes were observed under these conditions.

Should the two arcs come in contact with each other, a very loud noise is at once produced. The arcs often take the form as shown in *d*, Fig. 3, blowing each other out to one side to such a distance that the arc becomes ruptured. At other times they appear like jets of water meeting in midair, splashing and playing out into a fan-shaped mass of flame.

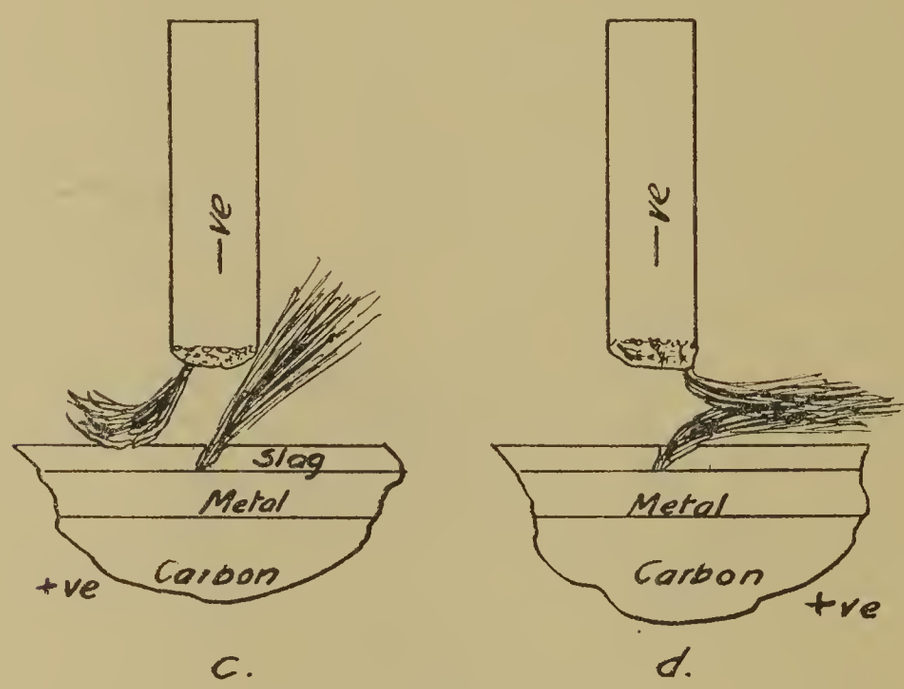


Fig. 3.

The attempts made to obtain photographs of these phenomena were not very successful, due to the rapid movement of the arcs.

During the spring of 1920 a certain amount of work was done at the FitzGerald Laboratories. The main object of these experiments was to study the effect of automatic regulation on the furnace currents.

A single-electrode Siemens type furnace was employed, in which a charge of scrap iron was melted, an acid calcium silicate slag being used. The moving electrode was of graphite, 6 inches (15.2 cm.) in diameter, and the furnace was run at a normal load of 60 kw.

An oscillograph was connected to the bus bars leading to the

furnace in such manner as to obtain sufficient potential to operate the vibrator.

Both G. E. and Thury automatic current regulators were employed, and numerous films taken at slow speed when the regulators were operating both to lower and raise the electrodes.

Numerous films were taken to obtain the wave form, and apparently at certain periods of operation a set of distortions covering a certain number of waves occurs at regular intervals of time. Hunting, which has been a great fault of automatic regulation, occurred during several of the runs.

Using the method as described above for projecting the arc on a screen explained hunting in this case to a certain extent. The "arc" normally consisted of a set of small arcs around the periphery of the electrode. These small arcs surrounded a central mass of flame apparently without any special form. The small arcs had the same general shape as a direct current arc between electrode and slag, the movable electrode being the negative pole.

When the electrode was close to the slag a heavy individual arc appeared on the inner edge of the electrode. By inner edge is meant that edge on the inside of the current-conducting loop formed by the bus bars, furnace terminals, electrode, arc and bath.

The results of numerous observations might be summarized as follows: A disturbance takes place in the furnace running on normal load with an arc as already described when the electrode is near the slag surface, tending to increase the current. The regulator immediately starts to raise the electrode. The large arc maintains its original shape until the length becomes so great that it is ruptured. Immediately all the lesser arcs move away to the outer edge of the electrode, leaving about 75 percent of the surface of the electrode end bare. This causes a rapid and large decrease in the current flowing, and the regulator commences to lower the electrode.

These arcs do not spread across the surface of the electrode until the surface of the slag is almost reached. They then suddenly spread over the whole electrode surface, causing a sudden and large increase in current, and the whole cycle of events is repeated.

In a case like this the regulator is hardly to blame for hunting.

Sometimes this hunting lasts for a period of three to five minutes, and ceases as abruptly as it commences.

Provision is being made for conducting further experiments during the coming winter, by means of the arc projections and oscillograms, to obtain a closer and more intimate knowledge of hunting and the general behavior of the arcs in electric furnaces.

The wave form and arc image will be simultaneously projected on the same screen, and "movies" of the two will be taken, so that the arc form will be seen at the same time as the wave when the pictures are projected in the usual manner.

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## DISCUSSION.

CARL HERING[1]: I understand that in Figure 3 an alternating current was used; it looks as though the arc acted as a rectifier and split the alternating current into two direct currents. Do each of those two arcs show the current in them to be direct? Was this investigated?

F. A. J. FITZGERALD[2]: Answering Dr. Hering's question, we found that, as nearly as we could judge, making our observations on the ground glass and studying the oscillographs afterward, there was a very strong rectifying action.

C. P. MADSEN[3]: I would like to ask Dr. FitzGerald what voltages were used on the arcs?

F. A. J. FITZGERALD: In the experiments made at our place, I think 45 volts was used. It was an ordinary Siemens' furnace.

CARL HERING: Was the double arc phenomenon shown in Figure 3 ever found to exist with a direct current?

F. A. J. FITZGERALD: Not that I know of; it was never found to be the case with the direct current.

E. R. MORTON[4]: In reference to Figure 3, the drawing shows

[1] Consulting Electrical Engineer, Philadelphia, Pa.
[2] FitzGerald Laboratories, Inc., Niagara Falls, N. Y.
[3] Consulting Engineer, New York City.
[4] New York City.

a rather thin layer of metal over the carbon. If that is anywhere near to scale, it is possible the arc ruptured through the metal from the lower carbon electrode.

F. A. J. FITZGERALD: That is only a rough diagram; it does not show the proportion at all. I do not think there was any rupture of the metal in the experiment, not from the observations we made.

*A paper presented at the Thirty-eighth General Meeting of the American Electrochemical Society, in Cleveland, September 30, 1920, President Landis in the Chair.*

# HEAT LOSSES THROUGH ELECTRODES OF A SIX-TON HEROULT FURNACE.[1]

By M. R. Wolfe and V. de Wysocki.[2]

The furnace investigated was a six-ton Heroult electric steel furnace in operation at the plant of William J. Wharton, Jr., & Co., near Easton, Pa. The problem undertaken was to determine the heat loss of electrodes under actual working conditions.

To measure the temperature, a thermo-couple of nickel and iron wire was used, the ends being twisted and fused together and connected with the voltmeter. The thermo-couple was standardized by use of an electric muffle furnace in conjunction with a standard platinum-iridium thermo-couple. The standard curve thus obtained was used for measuring the temperatures of the different parts of the electrodes, the junction being pressed against the electrode by means of a pad of asbestos on the end of an iron rod.

The six-ton furnace was provided with three electrodes, each 38 centimeters in diameter, of ordinary amorphous carbon. In working up the calculated heat flow, Hansen's value for thermal conductivity was used, *viz.*, 0.016 calorie per second per centimeter cube per degree centigrade.

Each electrode was provided with a water-cooled ring immediately above its exit through the roof of the furnace; also with a water-cooled connection at varying distances above the roof of the furnace. (See Fig. 1.)

## *Heat Losses in Cooling Water.*

The following tests of each of the three electrodes, determining the amount of water supplied to each electrode and its rise of tem-

[1] Manuscript received July 12, 1920. Abstract of thesis for degree of Electrometallurgist at Lehigh University.

[2] Candidates for degree of Electrometallurgist, Lehigh University.

perature, enables us to calculate the electric energy represented by the heat in cooling water used by each electrode:

| | Electrode | | |
|---|---|---|---|
| | 1 | 2 | 3 |
| Temperature of incoming water, C. degrees... | 12 | 12 | 12 |
| Temperature of outflowing water, C. degrees.. | 33.5 | 33 | 38.3 |
| Rise in temperature, C. degrees..... | 21.5 | 21 | 26.3 |
| Grams of water flowing per second..... | 427.8 | 427.8 | 427.8 |
| Calories per second ..... | 9,198 | 8,984 | 11,251 |
| Kilowatts absorbed ..... | 38.4 | 37.6 | 47.1 |

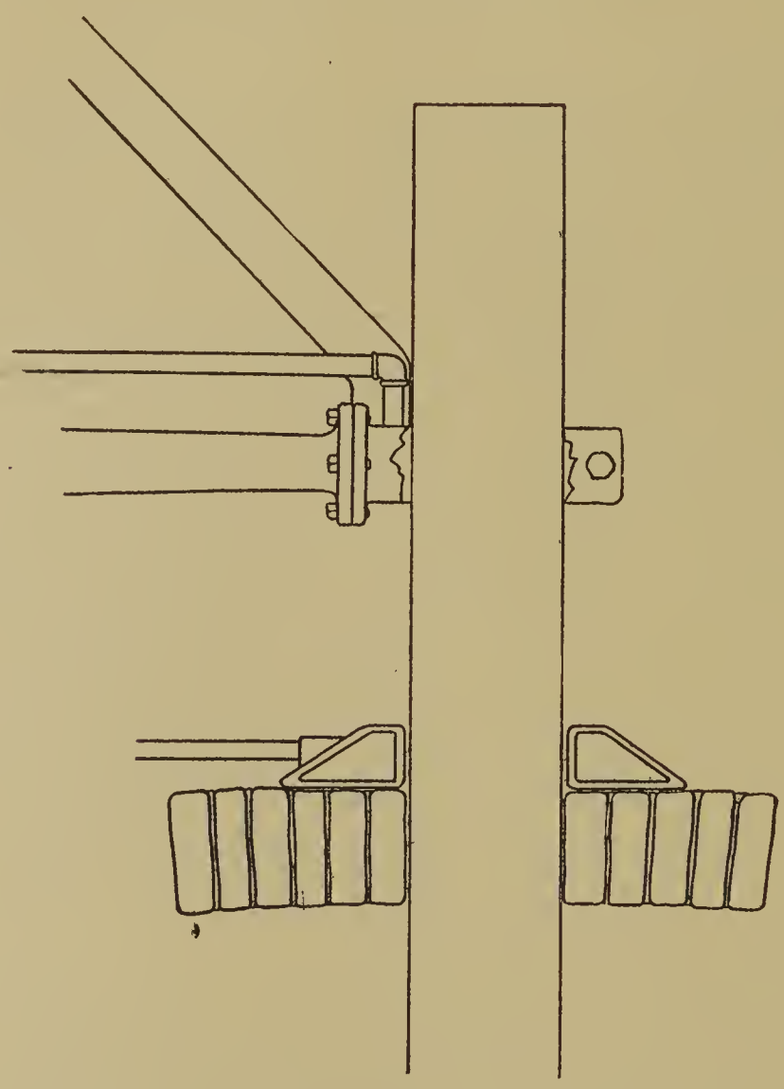

Fig. 1.

The sum total of heat in the cooling water for the furnace is, therefore, 123.1 kw. Since 650 kw. was supplied to the furnace, the total electrode loss is 18.7 percent of the power used.

In order to separate the heat carried by the cooling water of the upper cooling band from that of the lower band (the cooling water passed in series through the two bands of each electrode), an attempt was made to measure the heat flow into the upper

water-cooled connection, by noting the temperature of the electrode below this connection and above it, at a distance of 20 centimeters apart, and calculating the heat flow through these sections. Following are the data obtained:

| | Electrode | | |
|---|---|---|---|
| | 1 | 2 | 3 |
| Temperature difference, C. degrees | 156.7 | 138 | 188 |
| Thermal resistance of section of electrode | 0.1095 | 0.1095 | 0.1095 |
| Calories heat flow | 1,433 | 1,260 | 1,715 |
| Kilowatts | 6.0 | 5.3 | 7.2 |
| Kilowatts for lower band (difference) | 32.4 | 32.3 | 39.9 |

It thus appears that a total of 18.5 kw. was absorbed by the upper cooling contact holders, leaving 104.6 kw. absorbed in the lower water-cooled rings.

A small amount of heat is radiated from the electrode between the upper and lower cooling bands, but it is negligibly small in comparison with the heat carried off by the cooling water.

In conclusion, we wish to point out the lack of satisfactory data for the heat conductivity of carbon electrodes of different diameters and at different temperatures. There would seem to be a great field for experimental work in this direction.

We wish to record our obligations to Mr. A. S. Hummell, of the William J. Wharton Steel Works, for permission to make the tests.

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## DISCUSSION.

F. A. J. FitzGerald[1]: Is it fair to call these losses that were measured, electrode losses? Is not some of the heat lost from the roof? Those cooling rings as shown in the figure are, I think, put on very largely for the purpose of keeping the bricks around the electrode cool, and not so much with the idea of cooling the electrode, so that a certain amount of heat that is lost undoubtedly comes by conduction through the roof, and not from the electrodes, and therefore ought not to be called electrode loss.

[1] FitzGerald Laboratories, Inc., Niagara Falls, N. Y.

CARL HERING[2]: It is a pity the authors did not state the current also, so that it might be possible to use their figures for making deductions on other sizes of electrodes. The electrode projected considerably above the upper water-cooling ring; unless that upper water-cooling ring cools the electrodes to the temperature of the air, there will be a further loss above the upper ring, and that further loss is not accounted for in the results. Therefore, if that is the case, and no doubt it is, the actual loss is greater than 18.7 percent, except that, as Mr. FitzGerald just now said, the 18.7 percent may include some of the roof loss.

ROBERT TURNBULL[3]: I wish to confirm what Mr. FitzGerald has said in regard to the purpose for which these cooling rings are intended. They were never intended to cool the electrode; such a thing would be impossible, and especially by radiation. They would not even have much cooling effect if they were clamped tight to the electrode instead of being placed loosely around it. The purpose of these rings is to cool the roof, or rather the circle of bricks in the roof through which the electrode passes. Before cooling rings were used, only from five to seven days' life could be obtained from the rings of silica brick in the roof. After cooling rings were used, from three to six weeks' life was obtained, resulting from the cooling effect of the cooling ring. The main losses in temperature, and the resulting heating of the water in the cooling rings came from the roof itself, and from the flames and hot gases which pass between the cooler and the electrode, and I think if heat losses from the electrode itself were measured by some other means than by the increase of temperature in the water, where allowance has to be made for the heat given off by the flames, gases, heating from roof rings, etc., one would find that only about one-quarter of the losses are due to the electrode. I think this paper would better be named *"Losses Caused by the Passage and Radiation of Heat Through and From the Roof of an Electric Furnace,"* because if the electrode did not go through the roof, there would be very little loss. The authors give 650 kw. as the amount of current

[2] Consulting Electrical Engineer, Philadelphia, Pa.

[3] Managing Director, United States Ferro-Alloys Corp., Niagara Falls, N. Y.

operating the furnace on which the tests were made. If the percentage of loss was 18 percent. with 650 kw. it would be much less with 1,500 kw., which is the amount generally used in a 6-ton furnace; in this latter, the losses, instead of being 18 percent, would be between 7 and 8 percent.

J. W. RICHARDS[4]: In my opinion the criticism is quite correct; the losses given are total losses, including that on the cooling rings, and are not all ascribable to the electrode itself.

COLIN G. FINK[5]: In reference to that conductivity value of 0.016 calorie per second per centimeter cube per degree Centigrade, Mr. Hansen determined that value fifteen years ago, and at that time we were getting pretty good electrodes, but I understand they are either better or worse today, so that the value *may* not apply to the electrodes which were used in the present experiment.

FRANK HODSON[6] (*Communicated*): This excellent little paper will, no doubt, surprise many electric furnace users as to the amount and cost of power they are losing through the furnace roof. After a close study of this particular problem for the last four years, the writer is only surprised that the losses recorded were not larger. If the amount of heat in the shape of flame that escapes from most furnace roofs were taken fully into account, the loss would be found to be nearer 30 than 18.7 percent. The method employed by the authors of the paper, whilst giving actual heat losses from the electrodes, only gives heat absorbed by the water cooler; a great deal more than this is usually lost.

In considering these heat losses, which perhaps would be better described as through the furnace roof rather than through the electrodes, it might be interesting to consider how the conditions governing the loss of heat and power can be improved. Broadly speaking, the causes are the heating up of electrode, due to (1) faulty electrode joint or defective electrode, (2) imperfect

[4] Prof. of Metallurgy, Lehigh University, Bethlehem, Pa.
[5] Head of Laboratories, Chile Exploration Co., New York City.
[6] President, Electric Furnace Construction Co., Philadelphia, Pa.

contact or badly designed electrode holders, (3) electrode penciling off at point of arc, (4) oxygen from the air combining with the carbon of electrode and burning off.

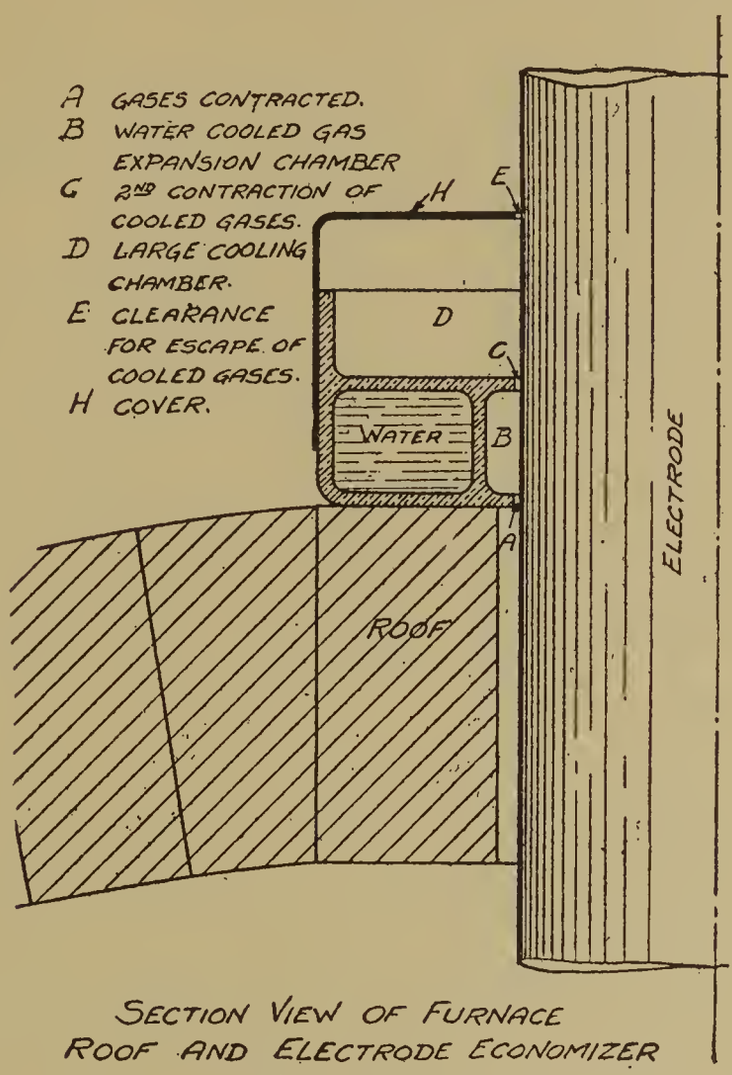
(1). Special care should always be taken in joining up electrodes; troubles due to this become increasingly worse as the melt proceeds. Good quality electrodes can now be purchased, and it pays to stand a higher price for proved electrodes.

(2). Design of electrode holder should be carefully considered and changed if undue losses occur. With proper contact, little or no heating should occur here. On our own furnaces we have not found it necessary to install water-cooled holders, looking upon these as an admission of failure to get proper electrical contact.

(3) and (4). *Penciling of Electrodes.* This is probably the indirect cause of more heat losses than any other feature. It is accentuated in cases where too large a space is left between the electrodes and the cooling ring, or where the furnace doors are not kept properly closed at the bottom, or where, as in some cases, the melter has to unduly open the doors to inspect or rabble the molten charge. The air strikes the white-hot electrode and burns away carbon, the hole in the roof acts as a flue, and the electrode pencils off, so that the area at the bottom, where the arc is, is often only one-fifth of what the furnace builders have designed to carry the power behind the furnace. The current continues to flow through this restricted area of electrode, and its resistance to the excessive current over the smaller area results in the whole electrode heating up, instead of this energy going into the arc. This also causes trouble with the joints, with the holders and on all the outside electrical connections. Sometimes large pieces or flakes of electrodes break off, due to the increased resistance.

With the above facts in mind, we have been working for the last five years to try and prevent this penciling and heating up of electrodes. It was realized early in the investigation that if the flame could be prevented from blazing out at the top that one of the chief troubles, *viz.*, penciling, would be largely over-

come. Experiments have been made with over a dozen different types of water cooler, and also attempts were made to insert an




SECTION VIEW OF FURNACE ROOF AND ELECTRODE ECONOMIZER

(Patent applied for)

FIG. 1. The Brooke Electrode Economizer.

inert gas under the furnace roof. As a result of these experiments, Mr. F. W. Brooke, Vice-President of the Electric Furnace Construction Co., has developed a type of electrode cooling ring or "Electrode Economizer," now fitted as a standard on all Greaves-Etchells Furnaces, that really does prevent this flame and consequent penciling of electrodes and other troubles caused thereby. These economizers have been fitted to a number of furnaces and have stood up remarkably well. Some have been in constant operation for six months, and are as good now as the day installed. The invention is covered by patents, but the company can supply these for all types of furnaces. A full description of this device was presented at the recent meeting of the Association of Iron and Steel Electrical Engineers, by Mr. E. T. Moore, Chairman of the Electric Furnace Committee,

pages 92 to 95, September, 1920, Report, but a brief description will probably interest A. E. S. members.

The Brooke "Economizer" depends for its action on the well-known scientific fact that hot gases under pressure, if suddenly allowed to expand, quickly lose their high temperature. The gases generated in an electric furnace are only combustible at high temperature and in the presence of oxygen. All other cooling rings simply content themselves with cooling these burning gases. In the Brooke Economizer the roof ports and the electrode are kept reasonably cool and the gases leaving the furnace are at too low a temperature to ignite. The gases pass first of all in between the electrode and the porthole roof, and then through the small clearance marked *A,* Fig. 1, in the cooling ring and into a relatively larger chamber, *B*, which causes the expansion of the gases and thereby giving up a large amount of sensible heat in the gases. This heat is absorbed by the water in the cooling ring. From chamber *B* they are once more contracted through the small clearance *C* and then passed to a very large chamber, *D,* for a second and chief expansion. The design differs slightly with amorphous or graphite carbon electrodes, but the effect is that no flame is seen from the top of the furnace and the electrodes do not pencil off. Even if the electrodes are withdrawn and the furnace is pouring, there is no flame or reduction of diameter. Nor does any flame appear when carbon is thrown on the slag. On a recently installed four-electrode furnace, the monthly figures were 12.5 lb. (5.7 kg.) of graphite electrodes per ton. The average over six months was 13 lb. (5.9 kg.). I should very much like the writer to make a series of similar tests on a furnace fitted with the special cooling rings. The electrodes in these furnaces never get red or white-hot outside the furnace, and it can be seen with the naked eye that a considerable saving of heat and power does take place.

J. W. Richards: I shall be pleased to have some of my students avail themselves of Mr. Hodson's offer, and make similar measurements on a furnace equipped with the Brooke economizer rings.

*A paper presented at the Thirty-eighth General Meeting of the American Electrochemical Society, in Cleveland, September 30, 1920, President Landis in the Chair.*

# HEAT CONTENT OF STEEL AND SLAG FROM AN ELECTRIC FURNACE.[1]

By A. M. Kuhlmann and A. D. Spillman.[2]

## Abstract.

Calorimetric determinations of the heat content of liquid steel and liquid slag from a six-ton Heroult steel-casting furnace. Also determinations of the heat capacity of the solid steel and solid slag. [J. W. R.]

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The following experiments were made on the heat content of the steel and slag produced in an electric steel furnace in operation at the works of the William J. Wharton, Jr., Iron and Steel Co., making steel castings from steel scrap melted in a six-ton Heroult electric furnace.

## APPARATUS USED.

Calorimeter was a galvanized iron bucket 21.6 cm. in diameter and 31.6 cm. high, insulated on the outside with a single layer of asbestos, 1 mm. thick. Calorimeter rested on a wooden stool; stirring was done by hand with a wooden paddle. Thermometer had a range of 50° C., was graduated in tenths, and temperature reading could be estimated to 0.01°.

For measuring temperatures a Fery radiation pyrometer was used for high temperatures and either a platinum-iridium thermocouple or platinum resistance pyrometer for the lower temperatures. The samples experimented upon in solid condition were brought up to heat in an electric resistance muffle-furnace.

[1] Manuscript received July 13, 1920. Abstract of thesis for the degree of Electrometallurgist at Lehigh University.

[2] Candidates for degree of Electrometallurgist, Lehigh University.

## DETERMINATION OF WATER VALUE OF CALORIMETER.

Water value of the calorimeter was determined experimentally by the method described by J. W. Richards (Journal of the Franklin Institute, Aug., 1901).

The water values found for the calorimeter were as follows:

| | | |
|---|---|---|
| 1 minute | 359 | calories |
| 2 " | 402 | " |
| 3 " | 454 | " |
| 4 " | 496 | " |
| 5 " | 533 | " |
| 6 " extrapolated | 562 | " |

These values were used as explained in the article referred to.

## HEAT CONTENT OF SLAG.

### *Solid State.*

The slag from the furnace had the following approximate composition as given by the chemist at the works:

| | Percent |
|---|---|
| $SiO_2$ | 23.5 |
| S | 0.65 |
| P | 0.5 |
| $Fe_2O_3+Al_2O_3$ | 24.0 |
| CaO | 42.0 |

Experiments were first made upon heat content of this slag in solid state at temperatures up to 810°, as measured with the platinum resistance pyrometer. Five experiments gave the following data:

| | | |
|---|---|---|
| 497° C | 93.3 | calories per gram |
| 622° C | 124.2 | " " " |
| 638° C | 134.9 | " " " |
| 685° C | 144.6 | " " " |
| 810° C | 179.5 | " " " |

Discussion of the above results gives as mean specific heat S (t to 0) and for the heat content Q (t to 0) the following expressions for solid slag:

$$S_m \ (0\text{-}t) = 0.133 + 0.00011t$$
$$Q \ (0\text{-}t) = 0.133t + 0.00011t^2$$

### *Liquid State.*

Two experiments were made on liquid slag at temperatures of 1190° C. and 1250° C. (radiation pyrometer), with the following results:

Q (1190°C-0).....476 calories
Q (1250°C-0).....520.5 "

## LATENT HEAT OF FUSION.

The heat content of solid slag evalued for 1190° C. gives 330 calories and for 1250° C. gives 340 calories. The two values of the latent heat of fusion thus deduced would be:

476 — 315 = 161 calories
520.5 — 340 = 180.5 "

Of these two values the former is probably more nearly correct.

## HEAT CONTENT OF STEEL.

The steel varied in composition from 0.06 to 0.20 percent carbon and from 0.1 to 0.2 percent manganese. Its heat content was determined both in solid and in liquid state.

### *Solid State.*

Temperatures were determined by the thermo-couple or the resistance pyrometer. The heat content was determined at 12 different temperatures ranging from 455° C. to 970° C. The results were, within experimental errors, coincident with the values given by Pionchon and Richards in Richards' Metallurgical Calculations, Volume II.

### *Liquid State.*

Several determinations of the heat in the liquid steel as it flowed from the furnace were as follows, corrected to 0° C.:

311.5 calories
302.7 "
306.8 "
312.5 "
315 "
324.3 "
325.2 "

We estimate that at 1900° C. the heat content of steel is 325 calories, as probably the maximum for the temperature given.

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## DISCUSSION.

J. W. RICHARDS[1]: The authors not only measured the heat in the liquid slag and steel, but they also made some experiments on the heat capacity of the slag and the steel in the solid state. The calorimetric work was rather carefully done. The method of making corrections for the heat losses in the calorimeter were determined experimentally by a method I have used for a good many years and found to be very reliable. I think that when the materials dropped into the calorimeter they measured the true heat content within half of one percent. The two determinations of the amount of heat in the liquid slag gave 476 and 520 calories per kilogram.

CARL HERING[2]: When molten steel is dropped into water there is a lot of steam generated and that means the absorbing of latent heat. This would give rise to a large error in using a water calorimeter, for such high temperature material. It would be much better to place in the calorimeter a cup containing lead and pour the molten steel into this lead; then measure the temperature after it has become uniform in the steel, lead and water. A correction for the specific heat of the lead and the steel might be necessary for accurate measurements.

W. S. LANDIS[3]: There are a number of things in the paper that I think require a little clearing up. One of them is the question of these temperatures. I notice the radiation pyrometer was used for measuring high temperatures. Usually around the steel furnace the radiation pyrometer is quite sufficient for the furnace attendant, but when it comes to actual scientific data, it has a great many errors and weaknesses which render its reading somewhat doubtful for exact work.

[1] Prof. of Metallurgy, Lehigh University.
[2] Consulting Electrical Engineer, Philadelphia, Pa.
[3] American Cyanamid Co., New York City.

J. W. RICHARDS: My own opinion is that the radiation pyrometer temperatures, as stated, are far too low, and that is confirmed by the fact that they got 476 and 520 calories in the liquid slag, which is probably reliable; deducting the correction for heat in the solid slag at 1,250°, leaves the latent heat of fusion 160 or 180 calories. I think 1,250° is not taken high enough; the latent heat of fusion of the slag is probably in the neighborhood of 100 to 130 calories. The determination of the temperature of a slag flowing out into the open is defective by the radiation pyrometer because of the unknown or indefinite emissivity of the melted slag, and this is a factor which should be determined for us by some physical experimenter. The total thermal emissivities of the materials are needed in order to calculate from the black-body temperature, which the radiation pyrometer gives, the actual temperature of the material. I think, however, that the calorimetric value of the heat in the melted slag and melted steel are reliable, and these are, for practical purposes, what we want to know with regard to the furnace. These are actual data which will tell us what the heat content of the slag and the steel is, which enables us to size up the thermal balance sheet of the furnace.

W. S. LANDIS: I would like to ask Professor Richards if the value for slag, and, in particular, the values for steel given here, correspond for this electric furnace steel very closely with some of the older figures we have been in the habit of using, and which were derived some eight or ten years ago by some German metallurgical laboratory?

J. W. RICHARDS: These values are in general somewhat higher. The values given for liquid steel from an open hearth furnace are usually 250 or 275 calories, up to 300 as a maximum. These all went over 300; the temperature was probably higher than crucible steel and certainly higher than open-hearth steel. In regard to the steam generated, as queried by Dr. Hering, if there was heat lost by the vaporization of water not accounted for, these values would all be too low. I think the loss by pouring hot metal and hot slag into water is small. I came to that conclusion by making a great many experiments. I made experiments, for instance, where the slag was poured into a copper

container immersed in the water. The slag then cools very slowly, but we can get its heat content if we continue the experiment long enough. We can then find the heat lost by vaporizing water by comparing that with the heat obtained by dropping the slag into the water directly. This difference is small.

CARL HERING: What I meant was to pour the steel into the solid lead and let the lead take up the heat of the steel and then give it to the water, which it would presumably do slowly enough not to waste any heat in the form of latent heat in making steam.

W. S. LANDIS: A paper of this sort is an extremely important contribution just at this time. We have been dependent in this country for a number of years on tables of constants either of German or French origin. We must not lose sight of the fact that we are now trying to make up our own book of constants in this country, and we want all the data of this kind we can get, good, bad or indifferent, open or not to criticism. There is going to be a competent committee appointed to comb out this material, but it is very important that a society of this sort keep up its end of that book of constants by the determination of just such physical information as this. That book will not go to press for some time at the rate it is progressing, but we want to have as many data relative to the electrochemical industry in there as we can get. I want the members of the Society to assemble these data and give us papers on them, even if they consist of only two paragraphs, but present your figures so that we will get the matter published and get it before the committee handling these new tables of constants.

A. L. FIELD[4]: I would like to ask Dr. Richards how the steel was melted and what was the carbon content?

J. W. RICHARDS: It was a low carbon steel, used for soft steel castings, melted in the furnace from scrap and stated to have between 0.18 and 0.20 percent of carbon. There are now two electric steel plants near Easton, Pa., utilizing the large amount of steel scrap to be found in the Lehigh Valley, and both working with power derived from the Easton Light & Power Company,

[4] Res. Physical Chemist, Electro-Metallurgical Co., Niagara Falls, N. Y.

generated by steam, and probably paying about the same price for it, about 1 cent per kw. hour, when using it "off-peak." Both these plants are running successfully and making a profit without any reference whatever to water power.

*A paper presented at the Thirty-seventh General Meeting of the American Electrochemical Society, and called up for discussion at the Thirty-eighth General Meeting of the Society in Cleveland, September 30, 1920, President Landis in the Chair.*

# ELECTRIC FURNACE SMELTING OF MONTANA MANGANESE ORES.[1]

By E. S. Bardwell.[2]

## Abstract.

An account of the plant of the Anaconda Copper Company at Great Falls, Mont., for reducing the Montana rhodochrosite manganese ores chiefly from the Emma Mine in the Butte district. Detailed description is given of the furnaces, the electrode holders, and the operation of the furnaces. In a test made on a half-and-half mixture of ores from the Philipsburg and Butte districts the charges and products were carefully weighed and analyzed, and balance sheet is given covering a period of 274 hours running, and therefrom is deduced a heat balance sheet showing the available heat in the furnace and its distribution. The author calculates the net useful efficiency of the furnaces tested to have been 71.7 percent. Of the heat losses sustained 40 percent was due to radiation and 60 percent to the electrical losses in transformers, conductors, electrodes and contacts. The composition and properties of the slags are discussed; also volatilization losses and the possibility that some are due to formation of carbonyls of iron and manganese. Finally the details of the calculation of the charge are given.—[J. W. R.]

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During the year 1918[3] there was produced in the United States 304,366 long tons of manganese ore, containing over 35 percent manganese. Of this amount, Montana alone produced 199,932 tons, or over 65 percent of the total tonnage. Practically all of this ore came from the Philipsburg and Butte districts;

[1] Manuscript received April 6, 1920.

[2] Metallurgist, Anaconda Copper Mining Co., Great Falls, Montana.

[3] The Mineral Industry (1918), 27, 460.

127,415 tons from the Philipsburg district, 72,381 tons from the Butte district, with only 136 tons reported as coming from other parts of the State. The armistice caused an immediate curtailment of manganese mining in Montana, owing to the ability of the eastern steel plants to again secure supplies of manganese ore from foreign sources.

Prior to the year 1914 less than 2 percent of the total high-grade manganese ore used in the United States was mined in this country. The manganese production of the State of Montana in 1918 was greater than the total imports to this country for the year 1911, and would represent a substantial percentage of this country's normal requirements.

In view of these facts, the importance of this potential source of manganese cannot fail to be recognized, and a discussion of the problems arising in the smelting of these ores in the electric furnace should be of interest.

## Character of Ores.

The Montana ores are low in phosphorus and iron and admirably suited for the production of 80 percent ferro-manganese.

In the Butte district the Emma Mine is the chief producer. The output of this mine during the war reached a maximum of 10,000 tons of rhodochrosite ore a month, the greater part of which was shipped to eastern consumers.

A typical analysis of this ore would be as follows:

| | Percent |
|---|---|
| Moisture | 2.61 |
| MnO | 46.87 |
| FeO | 2.68 |
| $SiO_2$ | 6.19 |
| $Al_2O_3$ | 1.18 |
| CaO | 0.00 |
| MgO | 2.61 |
| BaO | 0.00 |
| $CO_2$ | 33.56 |
| | 95.70 |
| Undetermined | 4.30 |
| | 100.00 |

The Philipsburg ores differ from those of the Butte district in that they are higher in silica and contain manganese principally in

the form of the psilomelane, a hydrated oxide of manganese of more or less indefinite composition.

An average analysis of this ore would be about as follows:

| | Percent |
|---|---|
| Moisture | 9.73 |
| $MnO_2$ | 50.10 |
| MnO | 3.83 |
| $Fe_2O_3$ | 2.90 |
| $SiO_2$ | 18.07 |
| $Al_2O_3$ | 2.01 |
| CaO | — |
| MgO | — |
| BaO | — |
| Loss on Ignition | 12.80 |
| | 99.44 |
| Undetermined | 0.56 |
| | 100.00 |

By washing, it is possible to reduce the silica content of the Philipsburg ores to at least one-half the percentage stated above, with a corresponding increase in manganese content.

## Electric Furnace Plant at Great Falls, Montana.

During the year 1918 the Anaconda Copper Mining Company erected at Great Falls, Montana, an electric furnace plant capable of producing 90 tons daily of 80 percent alloy. The first of five furnaces was started in September. Three furnaces were ready for operation at the time the armistice was signed, shortly after which time operation ceased, due to lack of market for the product.

The furnaces consist of an open-top, shallow brick shaft 28 x 14 x 9 feet (8.4 x 4.2 x 2.7 m.) in outside dimensions. The outer walls are of red building brick, with an inner firebrick lining. The shaft has a tamped lining composed of dead-burned magnesite, with pitch as a binder. The tamped lining is three feet (90 cm.) in thickness at the bottom of the crucible, and is brought up the sides to a point about 18 inches (45 cm.) above the tap-hole level. Cooling pipes are provided in the bottom, through which air for cooling is circulated. The inner dimensions of the crucible at the middle section are 22 x 9 feet (6.6 by 2.7 m.) by 6 feet (1.8 m.) deep. The furnaces are operated on 60-cycle, three-

phase alternating current. Two 24-inch (60 cm.) round amorphous carbon electrodes are used in a common holder on each phase. The electrodes are spaced three feet (90 cm.) between centers in the holders and the pairs about five feet (1.5 m.) on centers.

The holders originally provided were discarded on account of mechanical difficulties and another very simple holder devised. This holder is made in two entirely independent halves held together by pins and wedges. The long fish-tail cast integral with the holder makes it possible to avoid bringing cables out over the furnace, where they will be subjected to the hot gases. Water-cooling is brought about by a coil of pipe properly formed and cast in. The water connections for each half are independent. The holder is supported by yokes, which serve to keep the chains used for raising and lowering the holders up out of the heat. The winches for raising and lowering the electrodes are placed upon the bridge supporting the holder and electrodes, which bridge is insulated from the furnace superstructure by means of vitrified brick interposed between it and the super-structure. No trouble was experienced from failures of insulation during the time the furnaces were operated. The holders themselves were cast in sand molds from phosphor-copper, and required practically no machine work. We found the contact surface ample and the holder sufficiently flexible to take care of the inequalities in size often existing between the two electrodes in the holder. A loosely fitting sheet steel sleeve was provided to slip down over the electrode. This rested on top of the holder and prevented the electrode being eaten away above the holder by the hot gases.

When slipping electrodes the wedges were easily loosened, the holder raised and again clamped. The time required for slipping electrodes was not over ten minutes. No attempt was made to protect the electrode exposed between the holder and the charge, except to keep the holder as close down to the top of the charge as was feasible.

The sub-station from which electrical energy is supplied is located immediately back of the furnaces. It receives power at 110,000 volts from near-by hydroelectric plants of the Montana Power Company. Two 12,000 K. V. A. General Electric transformers serve to reduce the voltage from 110,000 to 6600 volts, at

which voltage it is delivered to the furnace transformers. Each furnace is equipped with one General Electric polyphase transformer rated at 5630 K. V. A., provided with taps on the high tension side for 85, 100, 115 and 130 volts. The furnaces were operated with a no-load voltage of 115 volts. The average input under these conditions was 3600 K. W. The power factor was about 80 percent.

The operating panels are arranged in a gallery overlooking the furnaces. Provision is made for either hand or automatic control. The automatic regulators are of the General Electric type originally designed to operate on current. We found it advantageous, however, to keep the voltage on the separate phases balanced rather than the current, and the necessary alterations were made in the apparatus to enable us to regulate on the voltage between phases and ground. With this change the automatic control proved highly satisfactory, holding the load constant under all conditions met with in operation with very little attention on the part of the operator.

The weighing and mixing division is so arranged that four operators can, in one shift, weigh and mix sufficient charge to keep five furnaces in operation for 24 hours. This would hardly be feasible were the plant treating a large variety of ores, but given a constant supply of ore of a uniform grade, little or no difficulty has been experienced. The mixed charge is delivered by belt conveyors to the mixed charge bin whence it is taken in boats by the crane and dumped into charge hoppers at the furnaces.

The charge consists of ore, lime-rock, coal, coke and iron ore. The only manganese ore treated at Great Falls is the Butte rhodochrosite ore from the Emma Mine. The charge treated was as follows:

| | | |
|---|---|---|
| Ore | 500 lb. | (227 kg.) |
| Lime-rock | 90 " | ( 41 " ) |
| Coal | 100 " | ( 45 " ) |
| Coke | 50 " | ( 23 " ) |
| Iron Ore | 12 " | ( 5.5 " ) |

When the furnaces were first started, coal only was used as a reducing agent. We soon found, however, that owing to the fact

that the rhodochrosite decrepitates when heated, this gave us too compact a charge. Primarily to do away with this difficulty, we substituted for a part of the coal an equivalent amount of blast-furnace coke based, of course, on the relative fixed carbon content of the two materials. With this change the furnace operated very smoothly, with only occasional boils. Coke alone was tried, but with this we could not keep the electrodes down in the charge. The proportions of coal and coke indicated above were finally determined upon as being the best under our conditions.

The furnaces are tapped at intervals of about 1½ hours. The slag and metal run into ladles, the slag is skimmed into the slag sluice and the metal cast by machine.

The plant was operated for such a short time and under such adverse conditions that it is impossible to give accurate metallurgical results. The results were on the whole, however, satisfactory. The loss by volatilization was not over 15 percent, and the recovery of manganese in metal between 75 and 80 percent.

## Electric Furnace Test on Montana Ores.

*Materials:* The ore used in the test about to be described consisted of a half-and-half mixture of ores from the Philipsburg and Butte districts. This material was very carefully bedded, sampled and analyzed. The analysis of the mixture gave results as follows:

| | Percent |
|---|---|
| Moisture | 5.03 |
| $MnO_2$ | 23.92 |
| MnO | 26.37 |
| FeO | 1.14 |
| $Fe_2O_3$ | 1.40 |
| $SiO_2$ | 13.48 |
| $Al_2O_3$ | 1.95 |
| CaO | 0.00 |
| BaO | 0.03 |
| MgO | 1.15 |
| $CO_2$ | 16.67 |
| Combined $H_2O$ | 6.43 |
| Undetermined | 2.43 |
| | 100.00 |

Coal was used as a reducing agent. This coal was of the following analysis:

| | Percent |
|---|---|
| $H_2O$ | 7.82 |
| Fixed carbon | 54.43 |
| Volatile combustible matter | 31.73 |
| $FeS_2$ | 1.84 |
| $SiO_2$ | 2.44 |
| $Al_2O_3$ | 0.73 |
| MgO | 0.34 |
| Undetermined | 0.67 |
| | 100.00 |

As flux, a very pure marble was employed. This contained 55.0 percent CaO and 43.2 percent $CO_2$.

It was necessary to add iron in order to make 70 percent ferro-manganese. The iron was added in the form of turnings, which were not analyzed, but which, for the purpose of the test, we assumed to contain 100 percent iron.

*The Furnace:* The furnace upon which this test was conducted was of the rectangular open-top type. It consisted of a steel shell 18 ft. x 9 ft. x 6 ft. 8 in. (5.4 x 2.7 x 1.9 m.) outside dimensions, with carbon lining approximately 30 inches (75 cm.) thick at the bottom of the crucible and 18 inches (45 cm.) on the side walls. The transformer equipment consisted of three single-phase Pittsburgh transformers, each rated at 667 K. V. A. and connected $\Delta/\Delta$. These transformers were provided with taps for 65, 75 and 85 volts, and during the test were operated at no-load voltage of 75 volts. Three 24-inch (60 cm.) round amorphous-carbon electrodes were used in water-cooled top holders. Motor-operated winches served to move electrodes up and down, but the furnace was not provided with automatic regulators. The furnace was tapped at intervals of about 1¼ hours. The slag and metal were allowed to flow together into sand pits, where they solidified and were then removed to the yard, where the slag was separated and metal weighed and loaded into cars for shipment.

*The Charge:* The furnace charge was varied slightly from time to time during the test, but was substantially as follows:

| | | |
|---|---|---|
| Ore | 500 lb. | (223 kg.) |
| Coal | 150 " | ( 68 " ) |
| Lime-rock | 90 " | ( 41 " ) |
| Turnings | 30 " | ( 14 " ) |

This charge gave us metal containing 70.9 percent manganese. The coal is, however, too high and the lime-rock too low for best results. A method for calculating the charge will be given later on.

*Balance Sheet:* The furnace was operated on the above charge for a period of 274 hours, during which time a total of 501,000 pounds (227,700 kg.) of the ore mixture was smelted, yielding 186,419 pounds (84,730 kg.) of metal containing 70.9 percent manganese. The slag from the furnace was kept separate from the slag resulting from other furnace operations, and was weighed, sampled, and analyzed. The complete balance sheet follows:

| Charge | | Metal | Slag | Gases |
|---|---|---|---|---|
| *Ore,* 501,000 Lb. | | | | |
| Mn | 178,096 | 132,171 | 4,482 | 41,443 |
| Fe | 8,831 | 8,831 | ..... | ..... |
| Si | 31,501 | 15,286 | 16,215 | ..... |
| $Al_2O_3$ | 9,790 | ..... | 8,342 | ..... |
| BaO | 150 | ..... | 925 | ..... |
| MgO | 5,753 | ..... | 4,313 | ..... |
| $CO_2$ | 83,517 | ..... | ..... | 83,517 |
| $H_2O$ | 57,415 | ..... | ..... | 57,415 |
| Comb. $O_2$ | 115,673 | ..... | 20,588 | 95,085 |
| *Flux,* 91,100 | | | | |
| CaO | 50,105 | ..... | 47,162 | ..... |
| $CO_2$ | 39,355 | ..... | ..... | 39,355 |
| *Coal,* 147,890 | | | | |
| Fe | 1,270 | 1,270 | ..... | ..... |
| Si | 1,688 | ..... | 659 | 1,029 |
| $Al_2O_3$ | 1,088 | ..... | ..... | ..... |
| MgO | 257 | ..... | ..... | ..... |
| Fixed C. | 80,495 | 9,135 | ..... | 71,360 |
| V. C. M. | 46,926 | ..... | ..... | 46,926 |
| $H_2O$ | 11,567 | ..... | ..... | 11,567 |
| *Iron Turnings,* 29,796 | | | | |
| Fe | 29,796 | 19,540 | ..... | 10,256 |
| Totals | 769,786 | 186,233 | 102,686 | 457,953 |

Metal and slag represent actual analyses.
2.97 percent only unaccounted for by balance sheet.

The analyses of metal and slag were as follows:

| Slag | Percent |
|---|---|
| Mn | 4.26 |
| $SiO_2$ | 34,37 |
| $Al_2O_3$ | 7.93 |
| CaO | 44.83 |
| MgO | 4.10 |
| BaO | 0.88 |
| | 96.37 |

| Metal | Percent |
|---|---|
| Mn | 70.9 |
| Fe | 15.9 |
| Si | 8.2 |
| C | 4.9 |
| | 99.9 |

| | | |
|---|---|---|
| Ratio ore to metal | 2.68 | |
| Percent total manganese in metal | 74.21 | |
| Percent total manganese in slag | 2.51 | |
| Percent total manganese in gases | 23.28 | |
| Percent of charge accounted for in metal | 24.22 | |
| Percent of charge accounted for in slag | 13.67 | |
| Percent of charge accounted for in gases | 62.11 | |
| Pounds slag per ton of metal | 1,129 | ( 513 kg.) |
| Pounds gas per ton of metal | 5,130 | (2,330 " ) |
| Pounds electrode per ton of metal | 150 | ( 68 " ) |
| K. W. hours per ton of metal | 4,879 | |

## Calculation of Theoretical Power Requirement.

From our balance sheet it is possible to compute a heat balance of the furnace. In so doing we shall assume

1. That the slag and metal contain at tapping 600 and 400 calories of heat respectively.

2. That the gases escape from the furnace at a temperature of 300° C.

The heat available is principally the heat of oxidation of the carbon, plus that furnished by the electrical energy used. The charge gives up to the carbon, as shown by the balance sheet, 95,085 pounds of oxygen. The 71,360 pounds of carbon burned would require 71,360 x 16/12 = 95,147 pounds of oxygen to burn to CO, or practically all of the carbon is burned to CO. Carbon monoxide is capable of reducing manganese dioxide to the monoxide, and it appears probable that a certain amount of reduction takes place in this manner. The balance of the carbon monoxide escapes as such and burns at the top of the charge.

The heat of formation of the slag will be taken as 150 calories per pound of contained $SiO_2$ and $Al_2O_3$. The heat of combustion of carbon with manganese is about 825 calories per pound of car-

bon combined, while the heat of formation of manganese silicide is 1693 calories per pound of silicon combined.

HEAT AVAILABLE

| | | Calories |
|---|---|---|
| Supplied by electric current | | $x$ |
| Oxidation of C to CO | = 71,360 x 2,430 = | 173,404,800 |
| Formation of silicate slag | = 44,500 x 150 = | 6,675,000 |
| Formation of $Mn_3$ C | = 9,135 x 825 = | 7,536,375 |
| Formation of $Mn_7$ $Si_2$ | = 15,286 x 1,693 = | 25,879,198 |
| | | 213,495,373 |

HEAT DISTRIBUTION

| | | Calories |
|---|---|---|
| Reduction of Mn from $MnO_2$ | 76,656 x 2,278 = | 174,622,368 |
| Reduction of Mn from MnO | 96,969 x 1,653 = | 160,289,757 |
| Reduction of Fe from FeO | 4,409 x 1,173 = | 5,171,757 |
| Reduction of Fe from $Fe_2O_3$ | 5,692 x 1,746 = | 9,938,232 |
| Reduction of Si from $SiO_2$ | 15,286 x 7,000 = | 107,002,000 |
| Calcination of $CaCO_3$ & $MnCO_3$ | 122,872 x 1,026 = | 126,066,672 |
| Distillation of volatile from coal | 46,926 x 800* = | 37,540,800 |
| Expulsion of moisture | 68,982 x 600 = | 41,389,200 |

* Assumed.

*Sensible Heat In Gases at 300°.*

| | | | Kg. Cal. |
|---|---|---|---|
| CO | 166,445 lb. = | 60,000 $M^3$ x 0.311 = | 18,660 |
| $CO_2$ | 122,872 lb. = | 28,148 $M^3$ x 0.436 = | 12,273 |
| $H_2O$ | 68,982 lb. = | 38,630 $M^3$ x 0.34 = | 13,134 |
| Vol. from coal | 46,926 lb. = | 21,285 $M^3$ x 0.311 = | 6,620 |
| Vol. manganese, etc. | 42,472 lb. = | 4,816 M x 0.8 = | 3,863 |
| | | | 54,550 |

54,550 x 300 = 16,365,000 Kg. Cal.

| | |
|---|---|
| Sensible heat in gases lb. cal. | 36,003,000 |
| Sensible heat in slag 105,201 x 600 | 63,120,600 |
| Sensible heat in metal 186,419 x 400 | 74,567,600 |
| Total heat energy required | 835,711,986 |

$$213{,}495{,}373 + x = 835{,}711{,}986 \text{ lb. cal.}$$
$$x = 622{,}216{,}613 \text{ lb. cal.}$$
$$1 \text{ K. W. hr.} = 1{,}897 \text{ lb. cal.}$$

hence required electrical energy, no allowance being made for radiation, is 328,000 K. W. hr.

During the test there were produced 93.2 tons of ferro-manganese. The theoretically required power is therefore

$$\frac{328{,}000}{93.2} = 3{,}520 \text{ K. W. hr., or say } 3{,}500 \text{ K. W. hr. per short ton.}$$

*Percentage Heat Distribution.*

| | Percent Total Heat Required |
|---|---|
| Reduction of manganese | 40.1 |
| Reduction of iron | 1.8 |
| Reduction of silicon | 12.8 |
| Calcination of $CaCO_3$ and $Mn\ CO_3$ | 15.1 |
| Distillation of volatile from coal | 4.5 |
| Expulsion of moisture | 4.9 |
| Sensible heat in gases | 4.3 |
| Sensible heat in slag | 7.6 |
| Sensible heat in metal | 8.9 |
| | 100.0 |

From the above it would at first appear that considerable saving might be effected by calcining the ore and lime-rock previous to charging the same into the furnace. This assumption, however, does not seem to be borne out in practice. The greater part of the work of calcination is brought about in the upper part of the charge column, utilizing heat which would otherwise be lost. Some economy of operation should, however, result, and the practicability of the operation should be carefully investigated.

From the above computation we are enabled to compute the heat efficiency of the furnace tested to have been 71.7 percent. This figure is very good for a furnace of the size upon which the test was conducted.

The heat loss is due, of course, in part to radiation of heat from the furnace itself and in part to $I^2R$ losses. A test carried out on a similar furnace showed 40 percent of the heat loss to be due to radiation and 60 percent to the $I^2R$ losses. Of this 60 percent the following distribution may be made:

| | Percent |
|---|---|
| Transformers | 17.5 |
| Conductors | 30.0 |
| Electrodes | 15.0 |
| Contacts | 37.5 |
| Total | 100.0 |

The above analysis gives us, I think, a very comprehensive idea of the heat requirements of an electric furnace producing ferro-manganese. Due to lack of accurate data certain assumptions had to be made in computing the theoretically required power, but the result obtained is sufficiently accurate for all practical purposes.

*Slags:* In studying electric furnaces producing ferro-manganese my attention was called to the great variety of slags produced.

It seemed worth while to investigate the question of slags with the idea in mind of seeing whether or not means could be found to calculate a charge which would give us a slag of low manganese content. From a considerable number of samples, eight typical slags were selected and carefully analyzed.

No. 1. A hard gray slag containing ferro-manganese inclusions.
No. 2. A medium green hard slag with few if any metal inclusions.
No. 3. A dark green hard slag with little in the way of inclusions.
No. 4. A high-lime air-slaking slag.
No. 5. A high-lime air-slaking slag.
No. 6. A light-grey glassy slag.
No. 7. A light-green hard slag.
No. 8. A light pea-green hard slag.

*Analyses of Slag Samples.*

| | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 |
|---|---|---|---|---|---|---|---|---|
| $SiO_2$ | 28.2 | 31.5 | 26.6 | 34.4 | 22.4 | 30.9 | 31.9 | 32.9 |
| FeO | 1.2 | 0.7 | 1.0 | 0.1 | 0.1 | 1.1 | 1.3 | 0.8 |
| $Al_2O_3$ | 17.3 | 8.0 | 9.6 | 7.7 | 6.8 | 10.0 | 10.1 | 8.7 |
| CaO | 41.4 | 35.7 | 31.1 | 42.2 | 46.8 | 37.8 | 35.3 | 36.0 |
| MgO | 7.6 | 1.8 | 1.1 | 1.8 | 4.0 | 10.2 | 10.4 | 2.1 |
| BaO | 0.4 | 1.3 | 4.8 | 1.1 | 0.2 | 0.2 | 0.3 | 1.2 |
| Mn | 2.1 | 14.5 | 19.9 | 8.1 | 5.3 | 4.7 | 5.8 | 12.2 |
| Total | 98.2 | 93.5 | 94.1 | 95.4 | 85.6 | 94.9 | 95.1 | 93.9 |

It will be noted that in this collection of slags we have two types only as regards their physical condition; *i. e.*, hard slags and air-slaking slags. The reason for the slags air-slaking is that the composition is such that calcium ortho-silicate, $2CaO.SiO_2$, separated as one of the phases. The ortho-silicate crystallizes in three distinct forms, the *alpha*-form, stable above 1410° C. to the melting point, the *beta*-form, stable between 675° and 1410°, and the *gamma*-form, stable below 675° C. The transformation of the *beta*-orthosilicate into the *gamma*-form is accompanied by a 10 percent volume increase. It is this volume increase, therefore, that accounts for what I have termed the air-slaking of slags 4 and 5. The presence of much magnesia in the slag tends to prevent this latter transformation.

Manganese occurs in the slags in three ways: First, as ferro-manganese inclusions; second, as manganese silicate; and third, as manganates or manganites of calcium or in some cases as oxides of manganese in solution in the slag. Any slag that is insufficiently

fluid will contain more or less in the way of ferro-manganese inclusions. Lack of sufficient carbon to reduce the manganese will, of course, promote the formation of manganates and manganites. Excess silica seems to tend to form silicates even when sufficient carbon is at hand to reduce all of the oxides of manganese to the metal.

A green slag is evidence of lack of sufficient carbon to carry out the reduction of the manganese oxide. The depth of color is an indication of the amount of manganese present. When there is an excess of silica present, slags are sometimes pink due to the presence of manganese silicate.

An excess of reducing agent tends to the formation of calcium carbide and graphite accretions in the furnace. This may in some cases be carried so far as to leave an excess of silica and promote the formation of manganese silicates in the slag; in any event, there is a waste of power and the accretions interfere with operations, rendering it necessary to shut down and dig out the furnace at frequent intervals. The best slags seem to have a composition of about 30 percent silica, 15 percent alumina and 50 percent lime plus magnesia, or better, should have an oxygen ratio, alumina being counted as base, of between 1.25 and 1.50. A slag of this composition is easily fusible, separates well from the metal and is economical to make.

*Volatilization Losses:* Under volatilization in the balance sheet has been included losses by dusting. In the electric furnace losses by dusting should not be large. Some loss by dusting is caused by occasional blows and boils occurring during periods of irregular operation. That the greater part of the manganese lost except for that contained in the slag is volatilized in some manner is very evident. In just what form or forms the manganese is volatilized I do not know, nor have I ever seen a satisfactory explanation of the phenomenon. It is usually considered that the manganese is volatilized as the metal, which, escaping from the furnace, burns at the surface of the charge to the oxide. The presence of brownish fumes in the gases escaping from the furnace would readily lead one to this conclusion. On the other hand, I have observed furnaces running with a comparatively cool top when there was no evidence of these brownish fumes, but which were still volatilizing a considerable amount of manganese.

In the case of a furnace volatilizing considerable amounts of manganese, I have observed that considerable amounts of iron, in fact based on the relative proportions of iron and manganese in the charge, more iron than manganese is volatilized.

An attempt to reduce the manganese content of the slag by using more carbon is accompanied in most instances by an increased volatilization loss.

In order that we may learn how to minimize our volatilization losses, it is important that we should know in what manner manganese is volatilized.

Excessive volatilization will occur inevitably in a furnace improperly designed as regards heat distribution. A furnace must be properly proportioned with reference to the power input in order to operate efficiently. Given such a furnace, however, we often find that the volatilization losses vary widely from time to time. The theory has been advanced that manganese and iron are volatilized as carbonyls, which in the hot-top furnace burn to the oxides of the respective metals and carbon dioxide and in the case of furnaces operating with a cooler top may pass off as gases unnoticed. This fits in very nicely with my observations with reference to addition of carbon, and its effect on increased volatilization. The formation of carbonyls, if such exist, may be due to the formation of carbides of iron and manganese, which in turn react with the metallic oxides present, reducing them and at the same time setting free carbonyls.

As in many cases, the slags produced by the electric manganese furnace have a distinct fluxing value. It might be well to throw more manganese into slag by cutting the carbon on the charge, provided the combined loss in manganese is not increased thereby.

*Calculation of Charge:* The charge will be calculated on the following basis:

1. We shall assume that the metal is to contain 70 percent manganese, 5 percent carbon, 5 percent silicon and 20 percent iron.

2. The charge will be calculated on the basis of 100 pounds of ore.

3. In proportioning the amount of reducing agent required, we shall assume that the fixed carbon only is available for bringing

about the reduction of the several metals. As has been pointed out, this is not a correct assumption, as some reduction is very evidently effected by the carbon monoxide gases and possibly the volatile hydrocarbons. It gives us, however, a starting point, and in the absence of complete data as to the reducing power of the fuel used, enables us to calculate the charge.

4. The magnesia will be calculated to $2MgO.SiO_2$, alumina to $Al_2O_3.3CaO.2SiO_2$ and the balance of the silica to $2CaO.SiO_2$.

5. Sufficient iron will be added to give 70 percent metal containing 20 percent iron, assuming 80 percent of the manganese to enter the metal.

*Carbon Requirement.*

| | | |
|---|---|---|
| C to reduce $MnO_2$ to Mn | $\frac{23.92 \times 24}{87}$ | = 6.60 |
| C to reduce MnO to Mn | $\frac{26.37 \times 12}{71}$ | = 4.46 |
| C to reduce FeO to Fe | $\frac{1.14 \times 12}{72}$ | = 0.19 |
| C to reduce $Fe_2O_3$ to Fe | $\frac{1.40 \times 36}{160}$ | = 0.31 |
| C to reduce $SiO_2$ to Si | $\frac{4.34 \times 24}{60}$ | = 1.73 |
| C to supply metal | | = 2.02 |
| | | 15.31 |

We shall, therefore, require $\frac{15.31}{0.5443}$ = 28.1 units of coal per 100 units of ore.

In the ore and coal we shall have to flux, after allowing for the 4.34 units of silica which is reduced and which enters the metal,

| | | |
|---|---|---|
| 9.82 | units | $SiO_2$ |
| 2.15 | " | $Al_2O_3$ |
| 1.24 | " | MgO |
| 0.03 | " | BaO |

Now figuring MgO to $2MgO.SiO_2$ we get

$$\frac{1.24 \times 140}{80} = 2.17\ 2MgO.SiO_2.$$

$Al_2O_3$ to $Al_2O_3.3CaO.2SiO_2$.

$$\frac{2.15 \times 388}{102} = 8.18\ Al_2O_3.3CaO.2SiO_2.$$

6.31 units of $SiO_2$ remains to be fluxed. Calculating to $2CaO.SiO_2$ we have

$$\frac{6.31 \times 172}{60} = 18.1\ 2CaO.SiO_2.$$

Total lime required = 15.24 units, or marble on charge would be

$$\frac{15.24}{0.55} = 27.7 \text{ lb. per 100 lb. ore.}$$

We have in slag

| | | Theoretical Analysis Percent |
|---|---|---|
| 9.82 | units $SiO_2$ | 34.4 |
| 2.15 | " $Al_2O_3$ | 7.6 |
| 1.24 | " MgO | 4.4 |
| 0.03 | " BaO | 0.1 |
| 15.24 | " CaO | 53.5 |
| 28.48 | | 100.0 |

Assuming 80 percent recovery of manganese, the iron required would be 6 lb. per 100 lb. ore.

Our charge, based on 500 lb. of ore, would then stand

| | | |
|---|---|---|
| Ore | 500 lb. | (227 kg.) |
| Coal | 140 " | ( 64 " ) |
| Lime-rock | 140 " | ( 64 " ) |
| Turnings | 30 " | ( 14 " ) |

If no special attempt is made to keep the silicon down to 5 percent, the lime can be cut to a considerable extent, depending upon the amount of silicon permissible in the metal. If low silicon metal is desired the coal may be cut, leaving the lime-rock as above. The coal may in any event be reduced somewhat, as has been pointed out previously, but this should be done a little at a time, and the furnace watched carefully. At least 24 hours should be allowed to elapse between such changes. The calculation of the charge can at best only serve as a guide. Much depends upon the characteristics of the furnace that is being operated. The operator must watch his furnace and vary the charge from time to time to suit operating conditions.

## DISCUSSION.

J. W. RICHARDS[1]: In the thermal balance sheet of the furnace, account is not taken of the volatilization of the manganese, and it is evident from the material balance sheet that a large amount of manganese was volatilized. I have calculated that it would represent about 3 percent of the energy of the current used in making the ferro-manganese. I would like to criticize the way in which the percentage of heat distribution is given on page 343. The distribution of electric energy is there given under about nine different heads, but that leaves out of account the volatilization of the manganese, also radiation and convection losses, and losses by conduction to the electrodes. One hundred percent ought to represent all the electric power used on the furnace, and also all the nine items into which we can divide the distribution of heat. Expressing as 100 percent the energy applied to the furnace, we would then have 30 percent of it used in the reduction of the manganese ore (instead of 40 percent) and so on correspondingly with each of the first nine items. The table ought to be thus revised to make it conform with the usual way of expressing the thermal balance sheet of the electric furnace.

On the next page an analysis of the slag samples is given and the percentage of manganese in them. I wish to call attention to the fact that the loss of manganese in the slag does not appear to be dependent on the percentage of the silica in the slags, nor to vary with the percentage of lime, so that neither the silica nor the lime determines the percent of manganese in the slag, but it is to be observed that the slags which contain the lower percent of manganese contain the higher percent of magnesia, and the four which contain the highest manganese contain the lowest magnesia. The four highest contain 13.8 percent. of manganese and 1.7 percent. of magnesia; it is therefore apparently true that by carrying high magnesia the percent. of manganese in the slags is reduced. There is, however, a still closer coincidence, *viz.*, that between the total percentage of alumina, lime and magnesia in the slag and the total percent of manganese; that slag

[1] Prof. of Metallurgy, Lehigh University, Bethlehem, Pa.

which has the highest percent of alumina, lime and magnesia has the lowest percent of manganese. All the slags arrange themselves without a break uninterruptedly in an inverse series; with the percentage of alumina, lime and magnesia from 66 to 41.8, the percentage of manganese in the slag runs uniformly from 2 up to 19. This gives the key to controlling the manganese in the slag, *viz.*, by varying the total percentage of alumina, lime and magnesia.

*A paper presented at the Thirty-eighth General Meeting of the American Electrochemical Society, in Cleveland, September 30, 1920, President Landis in the Chair.*

# THE SÖDERBERG ELECTRODE: FIRST AMERICAN INSTALLATION.[1]

By J. W. Richards.[2]

Abstract.

[Description of a three-phase installation of Söderberg continuous, self-baking electrodes, 32 inches (80 cm.) in diameter, in operation at Anniston, Ala., since July 2, making ferro-manganese. The furnace is 1800 K.W., and the operation is perfectly continuous as far as the electrodes are concerned.]

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"Elektrokemisk Industri" of Christiania, Norway, has made the first installation of this electrode in America, at the plant of the Southern Manganese Corporation at Anniston, Alabama. A description of the Söderberg electrode, which is continuous and self-baking, was read by the present writer at the Boston meeting of this Society, and is published in its Transactions.[3] The electrode therein described was a 34-inch (85 cm.) electrode, seen by the author in Norway, in the summer of 1919, where it was continuously operated in a ferro-silicon furnace from early in July until late in December, when it was shut down two days to give the workmen a Christmas holiday, and is still in operation. The furnace there operated had only one single-phase electrode, with bottom electrode in the hearth of the furnace to carry off the current.

Mr. Theodore Swann, president of the Southern Manganese Corporation, has recognized the great possibilities of this elec-

[1] Manuscript received July 23, 1920.

[2] Professor of Metallurgy, Lehigh University.

[3] Trans. Am. Electrochemical Society (1920), 37, 169.

trode, and has become the pioneer of its introduction into America. Other electric furnace plants will undoubtedly quickly follow his lead, as soon as their managers realize the great advance which it marks in electric furnace operation.

## THE FURNACE.

The furnace on which this installation is made is an open-top furnace of the usual ferro-alloy type. The working space is 12 by 20 feet (3.6 x 6 m.), elliptical in shape, and is normally run

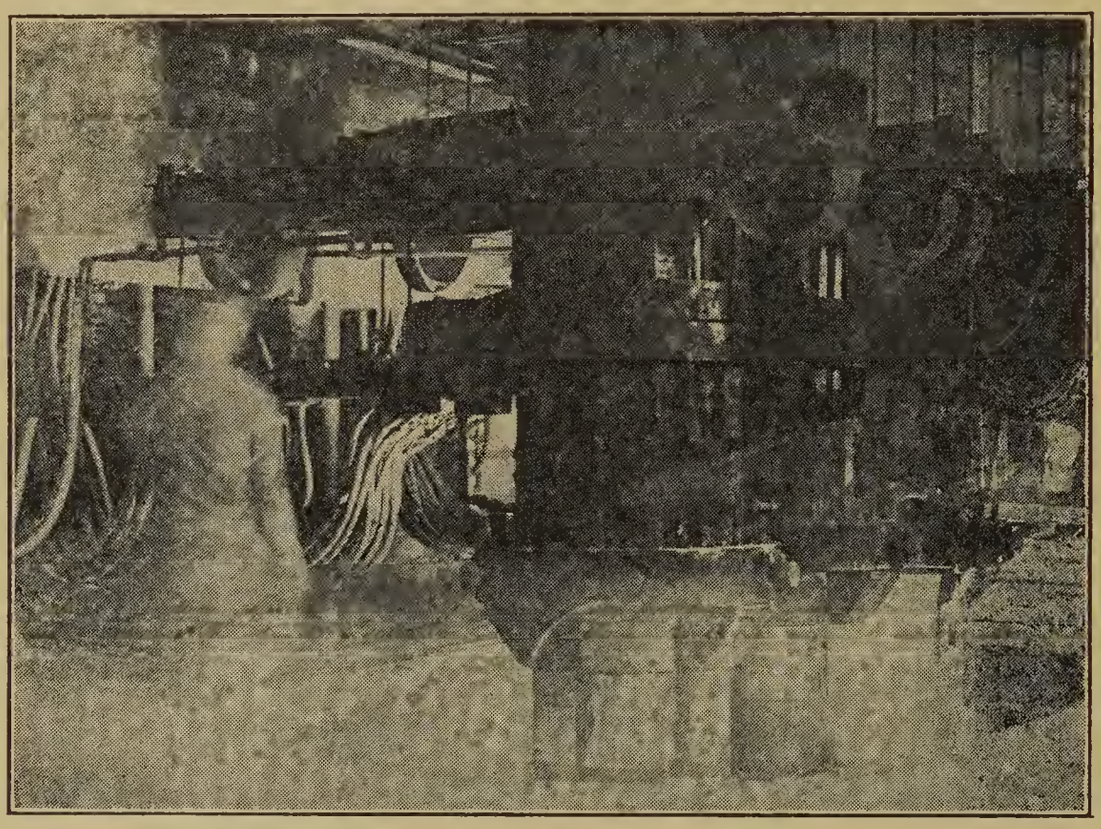

FIG. 1. Furnace empty, ready to run.

by three electrodes, each consisting of two 20 by 20 inch (50 x 50 cm.) baked electrodes, with top holders, in order to do away with screw connections. Such a furnace takes normally 1800 K.W., the three electrodes being on three-phase current, and spaced in line in the furnace. Slag and metal are tapped in the usual way. Primary current is received at 44,000 volts, 60 cycles, and is transformed in a three-phase General Electric Co. transformer to 68 volts, in delta connection on the low-tension side. The conducting cables, attached to the bus-bars passing through

the wall of the transformer house, are connected then to 4-inch (10 cm.) water-cooled copper pipes, which carry the current over the furnace to the holders. (See Fig. 1.)

## THE ELECTRODES.

The three electrodes are each 32 in. (80 cm.) in diameter, and extend from the furnace up into the tamping house, which is 12 ft. (3.6 m.) above the charging floor. The total length, when a new section has just been added to the top, is 24 ft. (7.2 m.), and the weight, exclusive of the holder, about 13,200 lb. (6000 kg.) per electrode.

The casing is formed of No. 18 sheet iron (1.05 mm.), riveted together to the correct diameter, and provided with inwardly directed partitions, as previously described. One section 46 in. (115 cm.) long weighs approximately 110 lb. (50 kg.), or 2.4 lb. per inch (0.44 kg. per cm.). Since the baked electrode mixture has a specific gravity of 1.5 and weighs 43 lb. per running inch (7.8 kg. per cm. of electrode) the iron weighs approximately 5.6 percent of the weight of the carbon electrode which it encloses, or 18 parts of carbon per 1 part of iron. Welding one section of casing into place takes 1 welder and 1 helper four hours. Tamping in of about 2000 lb. (900 kg.) of electrode mix, with pneumatic rammers, takes 2 men four hours, with one helper to carry mixture. A new section may be required per electrode every five to fifteen days, according to the nature of the ore being reduced in the furnace and the current taken by the electrode. In the straight-line three-phase arrangement, it is almost impossible to get each electrode to take the same current, so the consumption will be somewhat different on each electrode.

Holders and electrodes are suspended by wire cables controlled by hand in the controller house at the side of the furnace. (See Fig. 1.)

*Dust-shields.* Furnace dust is sure to settle on the electrode casing close to the furnace, and interferes with making good contact with the holder when the latter is raised. In the Norwegian furnace previously described, this was met by using a blast of compressed air to clean off the casing just above the

holder, immediately before raising the latter, and thus get a better contact. At the Anniston furnace this is obviated by providing a shield of heavy sheet iron which is attached to the upper part of the holder and encloses the electrode proper up into the dust-proof tamping house. This is permanently in place, effectually

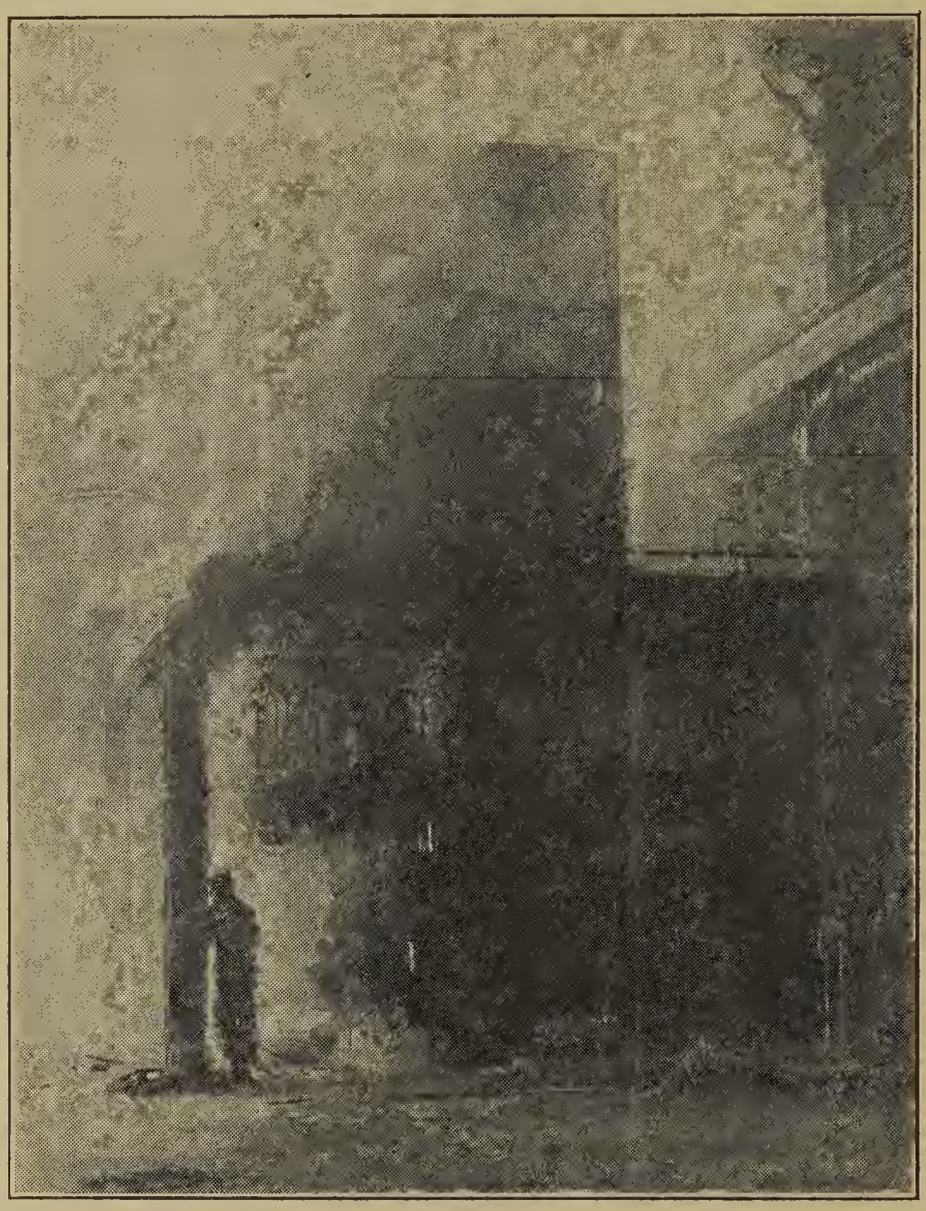

FIG. 2. Showing general structure, with tamping house above.

prevents dust from settling on the casing, and thus insures good contact for the holder when it is raised further up on the electrode.

*Holders.* While the design of these is immaterial to the functioning of the electrode, *per se,* yet the new holder constructed by engineers of the Norwegian company "Elektrokemisk Industri" is simple, rugged and effective. It is of the slip type and water-cooled. (See Fig. 3.) The screws are set by using a key with

an insulated socket, so that the men handling it on the charging floor get no ground currents. The virtual raising of the holder thus effected is done by three men in about three to four minutes, without taking current off the furnace. This operation is done for each electrode once or twice a day, with no interruption to the running of the furnace.

*Tamping house.* The booth for lengthening the electrodes is situated above the furnace; at Anniston it is 12 ft. (3.6 m.) above the charging floor and extends 11 ft. (3.3 m.) higher, having a clearance of 8 in. (20 cm.) below the traveling crane. Because of shortage of structural steel it was constructed of wooden scantling, covered with expanded metal and then with two inches of cement. Water-cooling pipes are placed in the floor, a passageway connects it with the outside of the building, and an exhaust fan keeps the air within it clear. (See Fig. 2.) Working in the tamping house is comfortable, the air being cooler and fresher than in the furnace room.

## WORKING OF THE FURNACE.

The ore used was Caucasian di-oxide ore of high grade, 56 percent manganese, with not sufficient iron for producing 78 percent ferro-manganese and not sufficient silica to give a proper slag volume in the furnace. Analysis of the ore showed 4.67 percent moisture. The dried ore contained:

| | | | |
|---|---|---|---|
| Manganese | 56.35 percent | Lime | 1.90 percent |
| Iron | 0.80 " | Magnesia | 0.84 " |
| Silica | 5.04 " | Baryta | 0.93 " |
| Alumina | 1.40 " | | |

| | |
|---|---|
| Manganese dioxide | 88.34 percent |
| Manganese monoxide | 0.65 " |

Iron turnings were added, and picked return slag put back in the furnace to increase the slag volume. The ore was so fine in structure that it packed badly in the furnace, causing frequent cratering. It was a difficult charge to work, requiring frequent stoking. Another furnace in the same plant, working the same ore with square baked electrodes, had the same difficulties from the packing of the charge. Considerable manganese was lost by

volatilization, because of the small volume of protecting slag on the metal bath and because of the frequent high-temperature outbursts of gases.

In 19 days' running, current was off the furnace 24 hours from

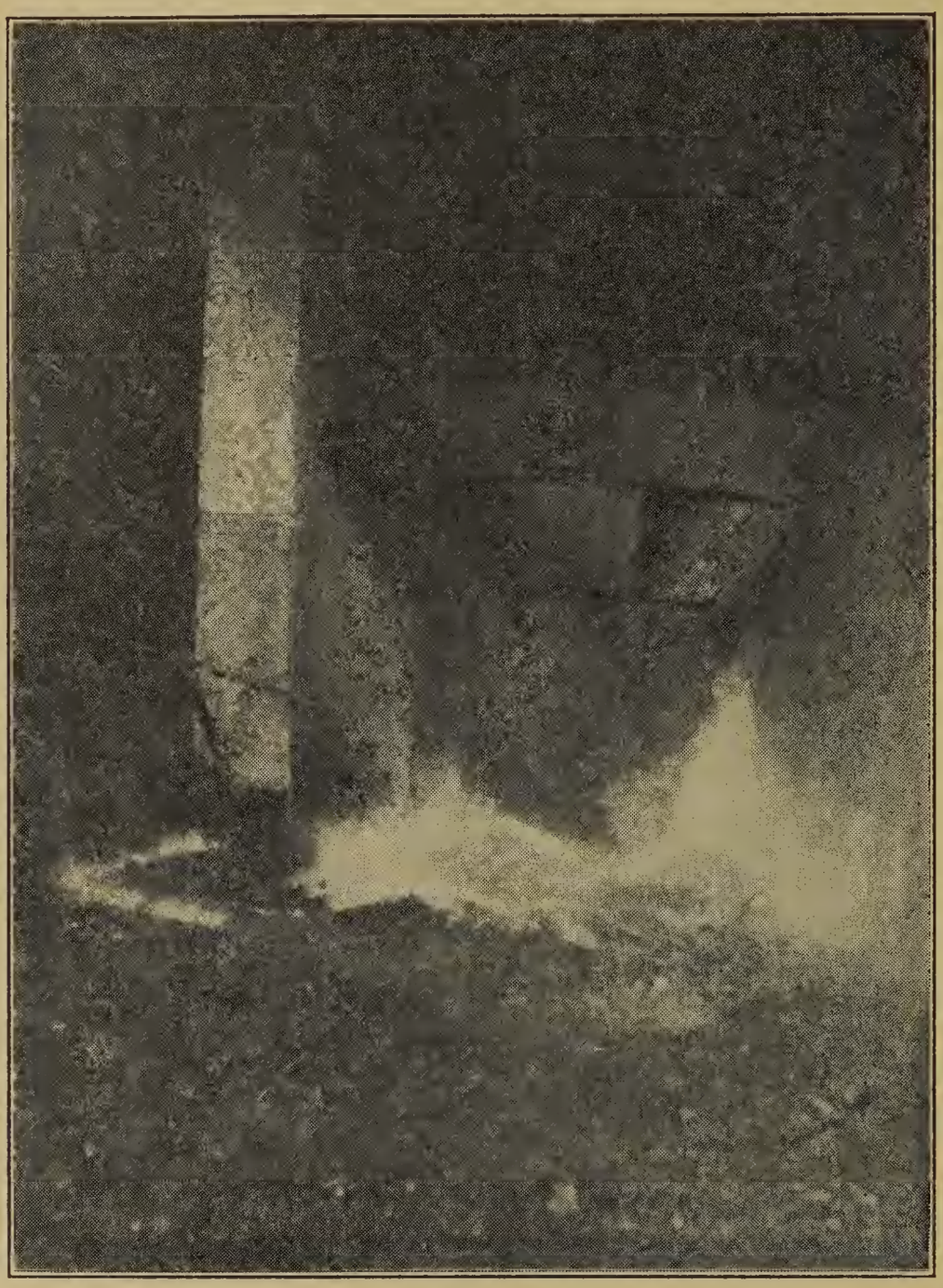

Fig. 3. Close-up of an electrode in action.

causes other than concerned the electrodes (such as lightning striking the transmission line, changing the voltage, trouble with oil switches, etc.), but was not interrupted one minute on account of the electrodes. In those 19 days the total electrode consumption amounted to 21 ft. 2½ in. (6.4 m.) on all three electrodes,

representing 10,600 lb. of baked carbon electrode consumed. During the same period 723,300 K.W. hr. passed through the furnace. The average carbon consumption was therefore 14.6 lb. (6.6 kg.) per 1000 K.W. hr. passing. This is considered a better method of expressing the carbon consumption than the usual K.W. hr. per ton of product, since the ore used was of exceptionally high grade, and the carbon consumption expressed per ton of product would seem unusually low.

The production of the furnace during this period was fully up to standard for the current used, and the consumption of carbon electrode was less than usual in working such rich ore. The labor required was that usual for a furnace of this capacity. There were no abnormalities of any kind either in the product, the slag, the labor required, or superintendence. On the other hand, there were no interruptions of current whatever on account of the electrodes, no poor joints, no breakages, no stumps, no "graveyard" for discarded electrodes.

The results of this furnace running prove the following advantages for the Söderberg electrodes:

1. Absolute continuity of operation, as far as the electrodes are concerned.

2. Smaller consumption of electrode carbon per given power supplied to the furnace.

3. Lower cost of electrode per given power supplied to the furnace.

4. Lower power losses in the electrodes.

5. Greater regularity of running of the furnace, and therefore less wear and tear on the workmen and the management, and greater uniformity of product.

In conclusion I wish to thank Dr. G. Jebsen, director of "Electrokemisk Industri" for permission to publish this description, and Mr. Westly and Mr. Corneliussen, engineers of the same company, for drawings and photographs of the plant which they designed and operated at Anniston. These engineers are part of the research staff of this company from its experimental plant at Fiskaa, Norway, where 2000 H. P. is utilized for experimen-

tal purposes. Licenses for the operation of Söderberg electrodes have already been given in Norway, France, Germany, South Africa and the United States.

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## DISCUSSION.

J. W. Richards: I will add a few facts which will bring the matter a little more up to date. During August, 1920, in 28 days, the furnace described was shut down only 13 minutes on account of the electrodes. The consumption of carbon electrode during those 28 days of August was 14.2 pounds per 1,000 kw. hours. A ferro-silicon furnace, which was in operation for a year in Norway, using Söderberg electrodes, had a consumption of 12 pounds of carbon per 1,000 kw. hours. The cost of the electrode at Anniston during August has been 4.12 cents per pound of electrode, covering all the cost and the license for using the process. As only one furnace is running, and the mixing apparatus and some other devices which are put in are capable of serving two or more furnaces, that cost will probably be lower when the electrode is used on more than one furnace. The cost of raw materials used was as follows: Anthracite, $18 per ton; coke, $14 per ton; tar and pitch, $25 per ton. Efforts have been made to adapt the electrode to the steel furnace. A recent cable from Norway says that 500 mm. (20 inch) electrodes have been used fourteen days on a Heroult electric furnace at the Stavanger Steel Works; they propose to put in two others, and cancel their orders for baked electrodes. I think it is probably quite practicable to apply the principle to aluminium production; the Norske A/S. for Electrokemisk Industri is working hard at their research plant to adapt it to that use.

F. A. J. FitzGerald[1]: Professor Richards mentions that there were lower power losses in the electrodes; how were these determined? If the power losses were distinctly lower in the elec-

[1] FitzGerald Laboratories, Inc., Niagara Falls, N. Y.

trodes using the Söderberg method, there should have been a considerable saving in power consumption in the plant; that is, an increased production per unit of power used, but Professor Richards says production was merely up to the standard. Now, if the losses of power did not occur in the electrodes, just where did they occur? Another question: have measurements of resistivity been made of these electrodes during the process of baking? They are of course presumably fully baked down at the tip, but up at the holders they are not so fully baked; have any determinations been made of how the resistivity changes? I think those would be interesting figures if Professor Richards has them.

ROBERT TURNBULL[2]: As a large consumer of electrodes, I am, of course, very much interested in this subject, and I think that if we can all make our own electrodes we will get away from the big profiteers. I have never seen the Söderberg electrode except in print, and as shown in the paper which Professor Richards has just read to us. In the latter, however, one cannot see anything but a blur, and whether it is an electrode or not, I do not know. It looks like one, but one has to draw on the imagination to believe it. But the paper Professor Richards has just read has changed somewhat the opinions I held after reading his first paper, last spring. One thing I noticed in that paper, if I may be pardoned for going back to it, was that the ferro-silicon furnace cited as using the Söderberg electrode in Sweden, which is single phase, used 11,000 kw. hours to make one ton of ferro-silicon. Average practice in this country at the present time shows that only from 5,500 to 6,000 kw. hours is required to make one ton of ferro-silicon; by ferro-silicon I mean the 50 percent grade, which I suppose was meant in the paper. Where did the other 5,000 go? Were they used to bake the electrode or what was done with them? I suppose the furnace was operated as well as furnaces are in this country. When the paper we have just heard came into my possession, I started a trial on some electrodes at a plant at Niagara Falls with which I am connected, using electrodes manufactured in this country, and we found the consumption of electrode to be 11.6 lb. per 1,000

[2] Managing Director, U. S. Ferro-Alloys Corp., Niagara Falls, N. Y.

kw. hours. Our test was conducted from August 25th until September 20th. We took three electrodes and put them one after the other in one of the phases of a three-phase furnace. When the first electrode was nine inches below the holder, we took the date and hour, and when the last electrode, or rather the one following on the last of the three, was nine inches below the holder, which showed that the three electrodes had been entirely consumed, a reading was taken of the number of kw. hours used during the test, and this gave us the consumption of electrode to each 1,000 kw. hours. This method of measuring the consumption of electrodes, by so many kw. hours, I do not think quite right, as a great deal depends on the method of operation, which can be changed to produce almost any given result. For instance, a ferro-silicon furnace operating with a minimum of carbon in the charge would have a much higher electrode consumption, because the electrode would work low down in the furnace below the reducing zone, and part of the silica and other matter would be reduced by the electrode. A reverse method of operating would be by the use of an excess of carbon, which would keep the electrode near the top of the charge, and in such a case the electrode consumption is lower. The latter is the best method of operating. What I would consider more reliable data, would be for Professor Richards to give us the pounds of electrode consumed per ton of product. I can give him our figures, taken from the test we made; they were sixty-five pounds. We used slightly less than 6,000 kw. hours to the ton of ferro-silicon throughout the test, and the amount of electrode consumed, to use Professor Richards' way of calculating, was 11.6 lb. to the 1,000 kw. hours. You may think I am knocking this thing, but having had such a long experience in electric smelting plants I am somewhat puzzled to understand how, in a modern plant, equipped with the best of modern mechanical appliances for handling raw materials to furnace, hot metals, etc., the Söderberg electrode could be used in conjunction with this other equipment. In our plant at the Falls we handle everything by overhead cranes. It takes five minutes to slip an electrode in the holder, and I do not think this can be considered a serious loss of time. Also I would like to say, where the current is not

taken off when slipping the electrode, as is apparently the case with the Söderberg electrode, I would consider such practice dangerous, as unless the Söderberg electrode has something mysterious about it, there is always the possibility of the electrode slipping in the holder and dropping to the bottom of the furnace, in which case a tremendous surge would follow, and possibly injure electrical apparatus. Taken altogether, I am not sufficiently convinced of the claims made in Professor Richards' paper on the Söderberg electrode to induce me to give it a trial.

F. R. KEMMER[3]: I would like to know what the resistivity of those electrodes is and how it has been measured?

W. S. LANDIS[4]: That question of the resistivity of these electrodes bothered me. I have not seen one in operation, but have had representatives, both abroad and in this country, study the working. The current is apparently carried by the sheet-iron jacket, so it does not matter at all what the resistivity of the mix is, at least from what I have been able to learn.

F. R. KEMMER: I notice there is a very low current density on all these electrodes, I think about 22 amperes to the square inch. I would like to ask Professor Richards whether higher current densities have been carried on this electrode than 22?

F. A. J. FITZGERALD: In reference to what Mr. Landis said, if the current is mainly carried by the sheet-iron covering of the electrodes, does that have any appreciable effect on the power factor of the electric circuit?

W. S. LANDIS: While I believe this electrode has some excellent possibilities, it was most unfortunate for its development, that the first installation was made so very far from the center of electric furnace activity. The Anniston installation is the only real large furnace plant in the South and is a long way from our Northern activity. Also the electric furnace for the production of ferro-manganese is hardly a typical example of general electric furnace practice, for the furnace operates at comparatively low temperature, has been developed satisfactorily only in small units, and in this particular case where custom smelting

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[4] American Cyanamid Co., New York City.

has been practiced is subject to very great irregularity in operation. As a result, I question if we are going to draw much valuable information from this Anniston installation. I believe the possibilities of the electrode would have been better developed had it been placed in some other class of electric furnace, and particularly one of somewhat larger power consumption.

J. W. Richards: Answering Mr. FitzGerald's question, I think the lower power losses in the electrode as determined, at least, down at Anniston, were due to the fact that the holder is very close down to the furnace on these new electrodes, and was on top of the electrodes before, so that there is less loss in the electrode. I was told that the power factor, as far as they could determine it, was slightly improved. The current density used was figured out to be exactly the same as they had been using on the other solid electrodes; exactly the same cross-sectional area in square inches was given to these three electrodes as was previously given to the square electrodes. The resistivity of the baked material averages the same as the ordinary baked electrode. Its appearance and ring is that of a first-quality baked material. It is true that the material does not bake until it is close down to the holder; a little way above the holder you can put an iron rod through the electrode, showing it is comparatively soft. The baking is almost all from the upper end of the holder down to the working end of the electrode. The current is carried from the holder to the iron casing and from that to the baked part of the electrode below the holder, and thence into the furnace. The consumption of carbon per ton of product is not put in the paper for this reason, that the manganese ore used was a very rich ore and the consumption of carbon per ton of ferro-manganese was so low that it looked abnormal. Another reason, the Southern Manganese Corporation said it was part of their business matters and they did not want it published. The slipping of the electrode with the current on has been successfully done for a year on the Norwegian ferro-silicon furnace. In the furnace down at Anniston they worked it successfully for about six weeks, but when attempting once to teach the other men around there how to do it themselves, the men loosened the

screws a little too far, and they had one slip of the electrode into the furnace.

Regarding power consumption per unit of ferro made. I am sure that this is not affected unfavorably by the Söderberg electrodes, but rather, favorably. The ferro-silicon furnace in Norway was of an old type and not supplied with the proper amount of current to work at high efficiency. In my opinion, other factors being alike, the furnace using Söderberg electrodes will work with lower power consumption per unit of product than when using baked electrodes of the usual kind.

My own impression of the furnace electrode situation is that this system of continuous self-baking electrodes is going to be applied wherever it is possible to put it in. It will accomplish, I think, a saving of about half of the present cost of using baked electrodes. As far as steel furnaces are concerned, that is going to be a very large field; while I have not the details of how this installation was made at Stavanger, the way in which they were talking about making it was to use short electrodes, not much longer than the present ones, and when the electrode became so short that it was down in the holder, lift it out, put it in a sand-box to keep it from burning, and there weld on the extra casing and tamp in the extra length of electrode, meanwhile replacing it at once by a longer electrode. In that way it would do away with all overhead construction, and the electrodes would be simply replaced by others as they were consumed. If it works that way, the system will be applicable at once to electric steel furnaces and several other types of furnaces.

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# THE ELECTRIC FURNACE AS APPLIED TO METALLURGY.[1]

## A READING LIST, 1900-1919.

By Clarence Jay West.[2]

The following pages contain a list of magazine references on the construction and operation of the electric furnace as applied to the metallurgy of iron and steel and the non-ferrous metals. An attempt has been made to include all the important references since 1900, at which time the electric furnace was becoming established in the iron and steel industry. Since 1907 the reference to Chemical Abstracts is given in addition to the magazine references, since this source will enable the reader to eliminate many of the references here given as unsuitable for his particular needs.

Criticism of the arrangement of this work is cordially invited, since we desire to make such studies of the greatest value to all interested.

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[1] Manuscript received February 9, 1920.

[2] Information Department, Arthur D. Little, Inc., Cambridge, Mass.

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## ABBREVIATIONS USED.

Am. Electrician ..... American Electrician
Am. Found. Assn. Trans..... American Foundryman's Association, Transactions
Am. Mach..... American Machinist
Ann. Soc. Ing. Arch. Ital..... Annali della Societa degli Ingegneri e degli Architetti Italiani
Appl. Sci. ..... Applied Science
Assoc. Iron Steel Elec. Eng... Association of Iron and Steel Electrical Engineers
Atti incoroggiamento Napoli.. Atti del r. istituto d'incoroggiamento di Napoli
Aust. Min. Stand..... Australian Mining Standard
Automotive Eng..... Automotive Engineer
Automotive Ind..... Automotive Industries

Berg. u. Hüttenm. Z..... Berg und Hüttenmännische Zeitschrift
Blast Furnace..... Blast Furnace and Steel Plant
Bol. soc. nacional minera Chile. Boletin de la sociedad nacional de minera de Chile
Bull. Am. Inst. Min. Eng..... Bulletin of the American Institute of Mining Engineers
Bull. Am. Inst. Mining Met. Eng. ..... Bulletin of the American Institute of Mining and Metallurgical Engineers
Bull. Can. Min. Inst..... Bulletin of the Canadian Mining Institute
Bull. sci. Liége ..... Bulletin scientifique, Liége
Bull. soc. inter. elec..... Bulletin de la Société Internationale d'Electriciens
Bull. tech. de la Suisse Romande..... Bulletin technique de la Suisse Romande
Bureau Mines Tech. Paper.... Bureau of Mines Technical Papers

C. A. ..... Chemical Abstracts
Can. Chem. J..... Canadian Chemical Journal
Can. Dept. Mines, Bull..... Canadian Department of Mines, Bulletin
Can. Elec. News..... Canadian Electrical News
Can. Eng..... Canadian Engineer, The
Can. Foundryman..... Canadian Foundryman
Can. Machy..... Canadian Machinery
Can. Min. J..... Canadian Mining Journal, The
Can. Min. Rev..... Canadian Mining Review
Can. Soc. Civ. Engrs..... Canadian Society of Civil Engineers
Cassier's Mag. ..... Cassier's Magazine
Chem. App. ..... Chemische Apparatur
Chem. Eng. ..... Chemical Engineer, The
Chem. Ind..... Chemische Industrie, Die
Chem. Met. Eng..... Chemical and Metallurgical Engineering
Chem. News ..... Chemical News
Chem. Trade J. ..... Chemical Trade Journal and Chemical Engineer
Chem. World ..... Chemical World
Chem.-Ztg. ..... Chemiker-Zeitung
Col. School Mines Mag..... The Colorado School of Mines Magazine
Compt. rend. ..... Comptes rendus hebdomadaires des séances de l'académie des sciences

| | |
|---|---|
| Durham Phil. Soc. | Durham Philosophical Society |
| Elec. Eng. | Electric Engineering |
| Elec. J. | Electrical Journal |
| Elec. News | Electrical News |
| Elec. Rev. | Electrical Review |
| Elec. Rev. West. Elec. | Electrical Review and Western Electrician |
| Elec. Times | Electric Times |
| Elec. World | Electrical World |
| Elecn. | Electrician, The |
| L'Elecn. | L'Electricien |
| Electrochem. & Met. | Electrochemist and Metallurgist |
| Electrochem. Ind. | Electrochemical Industry |
| Electrochem. Met. Eng. | Electrochemical and Metallurgical Engineering |
| Electrochem. Met. Ind. | Electrochemical and Metallurgical Industry |
| Elek. Kraft. u. Bahnen | Elektrische Kraftbetriebe und Bahnen |
| Elektrochem. Z. | Electrochemische Zeitschrift |
| Elektrotech. u. Polytech. Rundschau | Elektrotechnische und Polytechnische Rundschau |
| Elektrotech. Z. | Elektrotechnische Zeitschrift |
| Eng. | Engineer |
| Eng. Mag. | Engineering Magazine |
| Eng. Min. J. | Engineering and Mining Journal |
| Eng. News | Engineering News |
| Eng. Rev. | English Review |
| Eng. Soc. W. Penn., Proc. | Engineers' Society of Western Pennsylvania, Proceedings |
| Farben-Ztg. | Farben-Zeitung |
| Foundry Trade J. | Foundry Trade Journal |
| Gazz. chim. ital. | Gazzetta chimica italiana |
| Gen. Elec. Rev. | General Electric Review |
| Génie C. | Génie Civil |
| Giesserei Zeit. | Giesserei Zeitung |
| Glaser's Ann. | Glaser's Annalen |
| Glückauf | Glückauf, Berg-und Hüttenmännische Zeitschrift |
| L'ind. chim. min. met. | L'Industria chimica, mineria e metallurgica |
| Ind. electrique | Industrie electrique |
| Ind. Eng. | Industrial Engineering |
| Ind. Management | Industrial Management |
| Inst. E. E. J. | Journal of the Institution of Electrical Engineers |
| Inst. Min. Met. | Institute of Mining and Metallurgy |
| Intern. Cong. Appl. Chem. | International Congress of Applied Chemistry |
| Iron Coal Trades Rev. | Iron and Coal Trades Review |
| Iron Steel Mag. | Iron and Steel Magazine |
| Iron Trade Rev. | Iron Trade Review, The |
| J. Am. Inst. Metals | Journal of the American Institute of Metals |
| J. Am. Steel Treaters' Soc. | Journal of the American Steel Treaters' Society |
| J. Can. Min. Inst. | Journal of the Canadian Mining Institute |

| | |
|---|---|
| J. Chem. Met. Soc. S. Africa | Journal of the Chemical, Metallurgical and Mining Society of South Africa |
| J. Chem. Soc. | Journal of the Chemical Society |
| J. Clev. Eng. Soc. | Journal of the Cleveland Engineering Society |
| J. Elec. | Journal of Electricity |
| J. Elec. Power Gas | Journal of Electrical Power and Gas |
| J. d'Electrolyse | Journal de L'electrolyse et du four electrique |
| J. Eng. Club, Phila. | Journal of the Engineers' Club of Philadelphia |
| J. Eng. Club, St. Louis | Journal of the Engineers Club of St. Louis |
| J. four elec. | Journal du four electrique et de l'electrolyse |
| J. Franklin Inst | Journal of the Franklin Institute |
| J. Ind. Eng. Chem. | Journal of Industrial and Engineering Chemistry |
| J. Iron Steel Inst. | Journal of the Iron and Steel Institute |
| J. Royal Soc. Arts | Journal of the Royal Society of Arts |
| J. Russ. Chem. Soc. | Journal of the Russian Chemical Society |
| J. Soc. Chem. Ind. | Journal of the Society of Chemical Industry |
| J. West. Scotland Iron and Steel Inst. | Journal West of Scotland Iron and Steel Institute |
| J. West. Soc. Engineers | Journal of the Western Society of Engineers |
| Lumière elec. | La Lumière Electrique |
| Mach | Machinery |
| Manuf. Rec. | Manufacturers Record |
| Mech. Eng. | Mechanical Engineering |
| Mem. soc. ing. civ. de France | Memoirs de la Societé des Ingenieurs Civils de France |
| Met. et alliages | Metaux et alliages |
| Met. Chem. Eng. | Metallurgical and Chemical Engineering |
| Met. italiana | Metallurgia italiana, La |
| Metal Ind. | Metal Industry |
| Metall. u. Erz. | Metall und Erz. Zeitschrift für Metallhüttenwesen und Erzbergbau |
| Min. Eng. World | Mining Engineering World |
| Min. Ind. | Mining Industry |
| Min. Mag. | Mining Magazine |
| Min. Sci. | Mining Science |
| Min. Sci. Press | Mining and Scientific Press |
| Min. World | Mining World |
| Mining J. | Mining Journal |
| Mitt. kgl. Materialprüfungsamt | Mitteilungen aus dem königlichen Materialprüfungsamt zu Berlin Lichterfelde West |
| Mon. Sci. | Moniteur scientifique du Docteur Quesneville |
| Monit. tecn. | Moniteur Tecnico |
| Montan. Rundsch. | Montanistische Rundschau |
| Oesterr. Chem. Ztg. | Oesterreichische Chemiker Zeitung |
| Oesterr. Z. Berg. Hüttenw. | Oesterreichische Zeitschrift für Berg und Hüttenwesen |

Phys. Rev. .....Physical Review, The
Proc. Am. Inst. E. E.....Proceedings of the American Institute of Electrical Engineers
Proc. Am. Inst. Metals.....Proceedings of the American Institute of Metals
Proc. Eng. Club, Phila.....Proceedings of the Engineers' Club of Philadelphia
Proc. Inst. Civil Eng.....Proceedings of the Institution of Civil Engineers
Proc. Inst. Metals, London...Proceedings of the Institute of Metals

Rass. min. .....Rassegna mineraria, metallurgica e chimica
Rev. chim. ind.....Revue de chimie industrielle
Rev. d'electrochim.....Revue d'électrochimie et d'electrometallurgie
Rev. gén. chim.....Revue générale de chimie pure et appliquées
Rev. gén. sci.....Revue générale des sciences pures et appliquées
Rev. industriel .....Revue industrielle
Rev. de l'ingenieur et index techn. .....Revue de l'ingenieur et index technique
Rev. metal. .....Revue de Métallurgie
Riv. marit. .....Rivista Marittima
Rivista tecnica d. ferr. ital.....Rivista tecnica della ferroire Italiane
Roy. Soc., Phil. Trans.....Royal Society of London, Philosophical Transactions
Ry. Age .....Railway Age

Schweiz. Bau. .....Schweizerische Bauzeitung
Sci. Am. S. .....Scientific American Supplement
Sibley J. Eng.....Sibley Journal of Engineering
Stahl u. Eisen.....Stahl und Eisen

Tec. mod. .....La Technique Moderne
Tech. Paper .....Technical Papers
Trans. Am. Brass Founders' Assn. .....Transactions of the American Brass Founders' Association
Trans. Am. Electrochem. Soc.Transactions of the American Electrochemical Society
Trans. Am. Inst. Chem. Eng...Transactions of the American Institute of Chemical Engineers
Trans. Am. Inst. E. E.....Transactions of the American Institute of Electrical Engineers
Trans. Am. Inst. Metals.....Transactions of the American Institute of Metals
Trans. Am. Inst. Min. Eng....Transactions of the American Institute of Mining Engineers
Trans. Can. Min. Inst.....Canadian Mining Institute, Transactions
Trans. Eng. Soc. Toronto.....Transactions of the Engineering Society of Toronto
Trans. Faraday Soc.....Transactions of the Faraday Society
Trans. Inter. Eng. Congress...Transactions of the International Engineering Congress

U. S. Bur. Standards Bull.....United States Bureau of Standards, Bulletin

W. Engineering .....Western Engineering
W. Soc. Eng. J. .....Western Society of Engineers, Journal
West. Chem. Met.....Western Chemist and Metallurgist
Wis. Engr. .....Wisconsin Engineer

Z. angew. Chem. .....Zeitschrift für angewandte Chemie
Z. d. Oest. Ing. u. Arch. Ver...Zeitschrift des Oesterreichischen Ingenieur und Architekten Vereines
Z. Elektrochem. .....Zeitschrift für Elektrochemie
Z. Ver. deut. Ing.....Zeitschrift des Vereines Deutscher Ingenieure

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